APPENDICES

APPENDIX 6

STANDARD OPERATING PROCEDURE (SOP) GEHR3545

1.0 TITLE

General Electric (GE) Hudson River Design Support Sediment Sampling and Analysis Program Standard Operating Procedure for the extraction and cleanup of sediment/solid samples for Polychlorinated Biphenyl (PCB) analysis using the pressurized fluid extraction technique as per SW-846 Method 3545 for subsequent analysis by SW-846 Method 8082.

(Acknowledgement: This SOP is based substantially on internal method SOPs provided by Northeast Analytical, Inc. of Schenectady, N.Y.)

2.0 PURPOSE

The purpose of this SOP is to provide to the chemist the procedures required to perform extractions of PCBs, in sediment/solid samples, using the pressurized fluid extraction technique and to perform the subsequent extract volume reduction and cleanup for the GE Hudson River Design Support Sediment Sampling and Analysis Program.

3.0 SCOPE

The following procedure is utilized by the project laboratories for the extraction and cleanup of PCBs from sediment/solid samples using the pressurized fluid extraction method for subsequent analysis by SW-846 Method 8082.

4.0 COMMENTS

The soxhlet technique may be used in place of the pressurized fluid extraction at the discretion of the supervising chemist.

5.0 SAFETY

The chemist should have received in-house safety training and should know the location of first aid equipment and the emergency spill/clean-up equipment, before handling any apparatus or equipment. Safety glasses and protective exam gloves must be worn when handling glassware and samples. Polychlorinated biphenyls have been tentatively classified as known or suspected carcinogens. The chemist must review the Material Safety Data Sheets (MSDS) for PCBs and all reagents used in the procedure before handling them. All solvents should be handled within a lab fume hood.

6.0 REQUIREMENTS

The chemist must have an understanding of the methods and requirements of USEPA-SW- 846A "Test Methods for Solid Wastes" Volume 1B: Lab Manual, 3rd edition. Methods 3545, 3500B, 3620B, 3660B, 3665A. An approved instructor must also certify the chemist to perform the procedure.

7.0 EQUIPMENT

7.1 Cell Body: ASE 200™ (Accelerated Solvent Extractor) Dionex, 22ML #048821, 33 mL #048822 (or equivalent).

7.2 Cell caps: Dionex #049450 (or equivalent).

7.3 Steel Rod: Used to compresses sample in the cell.

7.4 Hydromatrix (Pre-cleaned and suitable for use): Varian #0019-8004 (or equivalent).

7.5 Metal spatula.

7.6 Mixing Tray: Used to mix sample prior to weighing sample.

7.7 Analytical Balance: Mettler AG-204 (or equivalent) used to determine sample mass.

7.8 Cellulose Filter: Prevents the frits of the cell end pieces from being clogged during ASE extraction.

7.9 Sodium Sulfate: Anhydrous (12-60 Mesh), washed with Hexane and baked overnight at 180°C. Used for the laboratory method blank.

7.10 Hexane: High Purity Solvent Baxter (Burdick/Jackson) #UN1208 (or equivalent).

7.11 Acetone: High Purity Solvent Baxter (Burdick/Jackson) #UN1090 (or equivalent).

7.12 Turbo Vap Evaporator: Zymark #ZW640-3 (or equivalent).

7.13 Turbo Vap Evaporator concentrator tubes: Zymark 250 mL (or equivalent), 0.5 mL endpoint.

7.14 1:1 Hexane/Acetone: 50%/50% by volume solvent mixture prepared in the lab.

7.15 Zymark Turbo Vap LV (or equivalent).

7.16 60 mL VOA vials.

7.17 Vials: glass, 8 dram & 4 dram (with Polyseal sealed cap) (20 mL & 10 mL) capacity, for sample extracts.

7.18 Vial Rack: Plastic rack used to hold vials, during all phases of the extract processing.

7.19 Centrifuge: International Equipment Co., Model CL (or equivalent).

7.20 Wrist Shaker: Burrell wrist action shaker, Model 75 and 88 (or equivalent).

7.21 Florisil: 10% deactivated, solvent washed with 1:1 hexane/ether, baked at 130°C for 16 hours. Deactivated with D.I. water. EM Science #FX0282-1 (or equivalent).

7.22 TBA Reagent: Tetrabutylammonium Hydrogen-Sulfite Reagent (prepared in the laboratory).

7.23 Mercury: Triple distilled Mercury Refining Co, Albany, NY #328502 (or equivalent).

7.24 Sulfuric Acid: H_2SO_4 (concentrated) Mallinkrodt #2468 #UN1830 (or equivalent).

7.25 Pipettes: S/P Disposable Serological Borosilicate Pipettes.

1. 1 mL × 1/10 #P4650-11X (or equivalent)
2. 5 mL × 1/10 #P4650-15 (or equivalent)
3. 10 mL × 1/10 #P4650-110 (or equivalent)

Kimble Pasteur Borosilicate glass pipette 9" #72050 (or equivalent)

7.26 Beakers: Assorted Pyrex: 250 mL, 600 mL, and 1000 mL.

8.0 PROCEDURES

8.1 Sample Preparation

8.1.1 Throughout the entire process it should be noted that if the chemist encounters any problems or difficulties with any samples or steps involved, these problems should be brought to the attention of the supervisor and/or quality assurance manager for guidance and then documented in the extraction logbook.

8.1.2 If the sample is a sediment and contains a water layer, decant and discard the layer as aqueous PCB waste. Mix the sample thoroughly and discard any foreign objects such as sticks, rocks, leaves, twigs, or pebbles. **Note:** however that the sample may be composed entirely of rock, concrete or some other solid material in which case the entire sample is treated as the solid.

8.2 Sample Extraction

8.2.1 Set up one 250-mL glass beaker or 4-oz. jar for each sample. Pick the first sample, label a beaker with the sample number, and tare the beaker. Using a metal spatula, add 10 g to 11 g of the wet sample to the beaker. Samples that are observed to be very wet will require additional mass of sample such that the project sensitivity requirements are met. The moisture content of the sample as determined in Section 8.2.2 should be evaluated so that a larger wet-weight sample can be obtained to provide a dry amount of solids to meet the project sensitivity requirements. The amount taken must consider the size limitations of the ASE extraction cell. The laboratory should target a wet-weight amount of 15 g for very wet samples. Record the weight in the PCB solid extraction logbook to the nearest tenth of a gram. Use the washed and baked sodium sulfate as the sample for the method blank and Laboratory Control Sample (LCS). Record the weight in the sample mass book.

NOTE: ALL SAMPLE CONTAINERS ARE TO BE RETURNED TO THE APPROPRIATE REFRIGERATOR. FOR ALL EMPTY SAMPLE CONTAINERS, SEE THE LABORATORY'S INTERNAL CHEMICAL HYGIENE PLAN FOR PROPER DISPOSAL.

8.2.2 The PCB concentration is to be determined on a dry-weight basis and therefore, the percent total solids must be determined. Weigh approximately 5 grams of the previously homogenized sample in a previously weighed, tarred aluminum-weighing pan. Record the weight of the sample and the tare weight of the pan in the percent total solids log. Place the sample in a drying oven at 100 to 110 degrees Celsius for at least 8 hours. Record the time placed in the oven and the oven temperature in the percent total solids log. Remove the samples from the drying oven and allow to cool in a desiccator. Weigh the pan and sample.

Calculate the percent solids by:

$$\frac{\{(\text{wt. of pan + dried sample}) - (\text{wt.of pan})\}}{(\text{wt. of wet sample})} \times 100\%$$

8.2.3 Before the sample is added to the cell, the sample must be dried. The sample is dried by adding pre-cleaned Hydromatrix. The amount of this drying agent being used depends on how much water is in the sample. The more water present in the sample, the more drying agent will be needed to dry the sample. Mix the sample and drying agent thoroughly with a metal spatula.

8.2.4 The cell size to be used during the extraction will be determined by the final volume of the sample after the drying agent has been added. **Note:** sometimes the sample will have to be separated into two cells for the extraction if to much drying agent has been added.

8.2.5 Select the appropriate cell body size for each sample. Assemble one cell end cap to the cell body. Place 3 cellulose filters into the open end of the cell and push it down to the cell end cap using the black ASE push rod.

8.2.6 Label cells with the sample number. Label the corresponding 60-mL VOA vials on the base of the vial.

8.2.7 Place the cell into a clean mixing pan. Add the dried extract to the cell using the metal spatula to guide the sample into the cell. Any sample that fell outside of the cell will be collected in the mixing tray. Remove the cell from the mixing tray and added the sample that is in the mixing tray to the cell. Compact the sample in the cell, using the steel rod, while the sample is being added. **Note**: rinse the steel rod with acetone and dichloromethane before using on a different sample or placing it in the storage drawer.

8.2.8 Add surrogate and matrix spike solution at this point. The final extract volume concentration of the surrogate compounds tetrachloro-*meta*-xylene (TCMX) and decachlorobiphenyl (DCB) should be 10 ng/mL and

100 ng/mL, respectively. At this time, the GE Hudson River Design Support Sediment Sampling and Analysis Program does not require the preparation and analysis of matrix spike and/or matrix spike duplicate samples. If requested in the future, the final extract volume concentration for the spiked Aroclor (Aroclor-1242) in the matrix spike and matrix spike duplicate sample should be 20,000 ng/mL (**Note:** this spike concentration will require a sample dilution to be performed). The final extract volume concentration of the LCS should be 500 ng/mL.

8.2.9 Assemble the top cell end cap to the cell, hand tighten. Place the first cell to be extracted in position 1 on the cell tray (top tray) and the 60-mL VOA vial in position 1 on the vial tray (bottom tray). The cells and 60-mL VOA vials for each sample must be in the same numerical position on the two trays.

8.2.10 Solvent used for PCB extraction is 1:1 hexane/acetone

8.2.11 Select the appropriate method or schedule for PCB extraction and start the ASE. Recommended ASE extraction conditions for PCB in Sediment are provided in Attachment 4.

8.2.12 When the extraction program is complete, transfer the hexane layer (top layer) using a 10-mL pipette into a pre-rinsed turbo tube or 60-mL VOA vial

if using Turbo Vap LV. Leaving only the water layer in the 60-mL VOA vial. Leave the 10-mL pipette in the turbo tube.

8.2.13 Rinse the 60-mL VOA vial using 5 pipettes of hexane. Hand shake for 30 seconds. Allow the two layers to separate, and pipette the hexane layer, using the same 10-mL pipette, into the turbo tube. Repeat this step 1 more time for a total of 2 hexane extractions on the water layer.

8.2.14 Rinse the 10-mL pipette with two pipettes of hexane on the outside of the 10-mL pipette that was in contact with the sample extract and two pipettes of hexane through the 10-mL pipette and collect into the turbo tube.

8.2.15 All glassware must be rinsed with technical grade (tech)-acetone or a "for rinsing-only" labeled solvent, and dried in the hood before other cleaning steps.

8.3 Solvent Reduction: TurboVap Evaporator System

8.3.1 The Turbo Vap evaporator system is used in place of the Kuderna Danish (KD)-concentrator apparatus. The turbovap uses a heated water bath and positive pressure nitrogen flow/vortex action. The unit maintains a slight equilibrium imbalance between the liquid and gaseous phase of the solvent extract which allows fractional reduction of the solvents without loss of higher boiling point analytes.

8.3.2 Turn the unit on and allow to heat up to the specified temperature for individual solvent use.

8.3.3 As a precaution the TurboVap system regulators should be checked to assure that no residual gas pressure remains within the system and that the gas cylinder valve and gas pressure regulators are both off before placing samples in the apparatus. Residual gas pressure may cause splashing and cross contamination of samples. To bleed the system of residual gas pressure place an empty turbo tube into the water bath and close the lid. Make sure that the nitrogen gas cylinder valve is turned off and slowly turn on the gas pressure regulator. Bleed any residual gas until the regulator output pressure gauge reads "0" psi. Proceed to 8.3.4. Make sure to wipe down all surfaces with hexane before concentration samples.

8.3.4 Place the turbo tube containing the samples into the TurboVap and close the lid. Turn on the gas cylinder valve first and then begin slowly turning the pressure regulator on.

Keep the gas pressure very low, until the solvent level is decreased, to avoid splashing. Increase the gas pressure as the sample reduces maintaining uniform flow throughout the reduction.

8.3.5 Turbo Vap – LV low volume unit: Turn the unit on and allow it to heat up to 38 degrees Celsius. As a precaution the Turbo Vap LV regulator should be checked to assure no residual gas remains in the system. Residual gas may cause splashing and cross contamination of the samples. To resolve this place a vial into the vial in to the turbo vap and close the lif. Press the start button and proceed to turn the gas regulator knob counter-clockwise until the regular reads zero. Place the 60-ml VOA vials into the turbo vap. Press the button to turn on the appropriate row of stations that are being used. The press the start button and adjust the regulator until the samples begin to swirl. Check the sample every few minutes and adjust the gas to keep the samples swirling.

8.3.6 The process for solvent (hexane/acetone) reduction takes approximately 20-30 minutes. Do not leave the unit unattended as extracts may be blown to dryness and PCB loss may occur. Immediately notify a supervisor if an extract is blown to dryness.

8.3.7 Concentrate the solvent to approximately 1.0 mL. Remove the samples from the TurboVap and place in the rack. The remaining solvent will consist largely of hexane since the acetone component is fractionally removed at a faster rate than hexane; however, a solvent exchange with hexane should be completed 3 times to ensure the acetone has been entirely removed. **Note**: Not all samples will evaporate at the same rate; sample extracts containing large amounts of petroleum or other non-volatile liquids may stop reducing

before the 1.0-mL point is achieved. Samples that stop reducing should be removed as soon as possible.

8.3.8 Quantitatively transfer the sample extract with a pasteur pipette into an appropriate volumetric flask (25 mL for soil extracts). Rinse the turbo tube or vial with 3 pasteur pipettes of hexane, then transfer the hexane rinse to the volumetric. Repeat the hexane rinse two more times for a total of three hexane rinses of the turbo tube. After the sample has been transferred, rinse the pasteur pipette with 0.5 mL of hexane into the volumetric flask. Add hexane to the volumetric meniscus mark.

Invert the volumetric flask at least three times to mix completely. Decant the contents into a pre-labeled 8-dram vial.

8.3.9 All dirty glassware must be rinsed with tech-acetone or a "For Rinsing-Only" labeled solvent and dried in the fume hood before being washed.

8.4 Sample Extract Cleanup

8.4.1 Most extracts of environmental samples that are to be analyzed for PCBs by gas chromatography with electron capture detection contain co-extracted xenobiotics and other interfering substances which must be removed before accurate chromatographic analysis can be performed.

8.4.2 Sulfuric acid, sulfur removal and Florisil® clean-ups should be performed on every sample. The sequence and number of replicates of cleanup steps performed are recorded by the sample preparation chemist on the sample tracking log sheet. Sample extract cleanups are performed on set volume extracts. The set volume is 25 mL for sediment/solid samples.

8.4.3 Sulfuric Acid Wash

8.4.3.1 The concentrated sulfuric acid treatment removes hydrocarbons and other organic compounds, which are co-extracted with the PCB residues.

8.4.3.2 Chill the sample to approximately 0°C. Add 5.0 mL of concentrated H_2SO_4 and shake for 30 seconds by hand, centrifuge for approximately 1 minute, transfer approximately 20 mL of the hexane upper layer to an 8-dram vial.

8.4.3.3 Repeat 8.4.3.2 if the sample extract appears to be heavily loaded (opaque) with colored material. Two to three acid washes may be required. **Note**: it is entirely possible that all colored material will not be removed from the extract.

8.4.4 Elemental Sulfur Clean-up

8.4.4.1 Elemental sulfur is soluble in the extract solvents used for sediment and soil samples. It is commonly found in many sediment/soil samples, decaying organic material, and some industrial wastes.

Large amounts of sulfur can cause the electron capture detector (ECD) to signal saturate for long periods during the elution envelope of PCBs. Even small amounts of sulfur can interfere with PCB measurement as a co-eluting chromatographic peak.

8.4.4.2 Two techniques exist for the elimination of elemental sulfur in PCB extracts. Mercuric precipitation (Mercury Shake) and the Tetrabutylammonium (TBA) sulfite procedure.

Tetrabutylammonium sulfite causes the least amount of degradation to a broad range of pesticides and organics compounds, while mercury may degrade organophosphorus and some organochlorine pesticides. The TBA procedure also has a higher capacity for samples containing high concentrations of elemental sulfur.

8.4.5 Removal of Sulfur Using Mercury

8.4.5.1 Mercury is a highly toxic metal. All operations involving mercury should be performed within a hood. Prior to using mercury, the chemist should become acquainted with proper handling and emergency spill/clean-up procedures associated with this metal and must have reviewed the material safety data sheet for mercury.

8.4.5.2 Add 1-3 drops of mercury to the sample extracts, cap, and place on the wrist shaker for 30 minutes. The sulfur is converted to mercuric sulfide and precipitates out of the sample extract. A black precipitate may be seen in sample extracts containing elemental sulfur.

8.4.5.3 Remove the sample extracts from the wrist shaker and place in the centrifuge at a setting and duration appropriate to spin down the solids.

8.4.5.4 Transfer the sample extract to a clean 8-dram vial.

8.4.5.5 The precipitated sulfur can be removed from the extract by performing a sulfuric acid clean-up or a Florisil® slurry.

8.4.6 Removal of Sulfur using TBA Sulfite

8.4.6.1 The TBA procedure removes elemental sulfur by conversion to the thiosulfate ion, which is water-soluble.

8.4.6.2 Add 2.0 mL TBA Sulfite Reagent, 1.0 mL 2-propanol, and approximately 0.65 g of sodium sulfite crystals to the extract and shake for at least 5 minutes on the wrist shaker and observe. An excess of sodium sulfite must remain in the sample extract during the procedure. If the sodium sulfite crystals are entirely consumed add one or two more aliquots (approximately 0.65 g) to the extract and observe.

8.4.6.3 Place the samples on the wrist shaker for 45 minutes observing at 15-minute intervals to make sure that the sodium sulfite is not consumed. Add 5 mL organic free water and shake for 10-15 minutes.

8.4.6.4 Place the samples into the centrifuge and spin at a setting and duration appropriate to spin down the solids.

8.4.6.5 Transfer the hexane layer to a new 8-dram vial and cap.

8.4.7 Florisil® Adsorption (Slurry)

8.4.7.1 The Florisil® slurry removes co-extracted polar compounds, residual water, and residual acid and is recommended as the final cleanup step before the extract is submitted for GC analysis.

8.4.7.2 Add approximately 3 grams of tested and approved 10% deactivated Florisil® to each vial containing the sample extract.

8.4.7.3 Vigorously shake the vial for approximately 1 minute by hand or on the wrist shaker.

8.4.7.4 Place the vial(s) into the centrifuge at a setting and duration appropriate to spin down the solids.

8.4.7.5 Transfer the extract to a clean 8-dram vial.

8.5 Extract Screening and Dilution

8.5.1 Screening of PCB extracts by GC to determine the approximate concentration before final analysis is highly recommended. If possible, prior site history and estimates of sample concentration will be provided by field personnel and may be used to determine what, if any, extract dilution is necessary.

8.5.2 The supervising chemist is responsible for determining initial screening dilutions. Extract dilutions are prepared by transferring an aliquot of the original sample extract into a vial containing the correct amount of "make up" volume of hexane. Dilutions must be recorded in the instrument logbook or in the data system.

8.5.3 Perform the dilution using an appropriate disposable volumetric pipette to transfer the extract and for the make-up volume of hexane. Make sure that the vial is properly labeled. Cap and invert the vial at least three times to thoroughly mix the extract with the solvent.

8.5.4 Transfer 1 mL of the extract to a labeled 1.5-mL GC autosampler vial. Record the sample data and submit with the sample extracts to the GC analyst.

9.0 QUALITY CONTROL

9.1 This section outlines the necessary quality control samples that need to be instituted at the time of sample extraction. The data from these quality control samples is maintained to document the quality of the data generated.

9.2 With each batch of samples to be extracted a method blank is processed. The method blank is carried through all stages of sample preparation steps (including clean-up steps). For sediment/solid samples, a laboratory sodium sulfate blank is processed.

9.3 At this time, the GE Hudson River Design Support Sediment Sampling and Analysis Program does not require the preparation and analysis of matrix spike and/or matrix spike duplicate samples. If requested in the future, a matrix spike for Aroclor-1242 is to be analyzed at a rate of 1 matrix spike per every 20 samples at a concentration of 20,000 ng/mL in the extract (**Note:** this spike concentration will require a sample dilution to be performed). Also a matrix spike duplicate sample is to be analyzed at a rate of 1 per every 20 samples.

9.4 A QC reference check standard (LCS) is also prepared and analyzed for Aroclor-1242 at a concentration of 500 ng/mL in the extract. For sediment/solid samples, sodium sulfate is used.

9.5 Surrogate compounds are added to each sample, matrix spike, matrix spike duplicates, method blank, and QC reference check standard (LCS) at time of extraction. The surrogate compounds TCMX and DCB are to be added for final extract concentrations of 10 ng/mL and 100 ng/mL, respectively.

10.0 REFERENCES

1. U.S. EPA SW-846 "Test Methods for Evaluating Solid Waste; Volume 1B Laboratory Manual Physical/Chemical Methods", Office of Solid Waste and Emergency Response, Third Edition, Final Update III, December 1996.

2. "Guide to Environmental Analytical Methods", Third Edition, Genium Publishing Corporation, 1996.

11.0 ATTACHMENTS

1. ASE Methods

2. ASE control panel keypad.

3. ASE cell cleanup procedure.

4. Recommended ASE Extraction Conditions for PCB in Sediment

ATTACHMENT 1

ASE METHODS

METHOD #	ANALYTE OF INTEREST	MATRIX	REQUIRED SOLVENT *	AMOUNT OF SAMPLE
1	PCB	WIPE	1	WIPE
2	PET I.D.	SOIL	1	10 g
2	8270	SOIL	3	30 g
3	**PCB**	**SOIL**	**2**	**10 g**
4	NONE	NONE	2	NONE
5	**PCB (RUSH)**	**SOIL**	**2**	**10 g**

*REQUIRED SOLVENT CHART

1 - HEXANE
2 - 1:1 HEXANE / ACETONE
3 - 1:1 DICHLOROMETHANE / ACETONE

ATTACHMENT 2
CONTROL PANEL KEYPAD

* TRAY	Tray is in free spin for manual turning.
TRAY *	Tray drive mechanisms are engaged and cannot be moved manually.
RINSE	Starts a manual rinse cycle.
* START	System is idle.
START *	system is currently running a method or schedule.
ABORT	Interrupts current run. Continue with abort function to terminate ASE run.
MENU	Displays a list of available screens.

* - LIGHT IS ON

ATTACHMENT 3
ASE cell cleanup procedure

Remove the end caps of ASE cells. Using a metal spatula designated for cell clean up, push the extracted sample out of the cell into a garbage can. Wash the interior and exterior of the cell and cell end caps with soap and water (use the brush designated for ASE use only). Dry the cell parts with a paper towel and reassemble the cell.

Run the washed cells on the ASE (use a new 60-mL VOA vial for each cell) using method 7 for 22 mL cells, 8 for 33 mL cells and 1:1 dichloromethane/acetone as the solvent.

Note: After the cells has been used 20 times or if the frits become clogged, the cell end caps should be taken apart and sonicated for 10 minutes in acetone and 10 minutes in dichloromethane.

ATTACHMENT 4
Recommended ASE Extraction Conditions for PCB in Sediment

The following instrument conditions will be utilized for extraction of sediment samples by accelerated solvent extraction for PCB. These conditions may need to be optimized, as needed, according to the instrument manufacturer's recommendations. Once conditions are established, the same procedures should be performed on all samples.

Recommended extraction conditions for Dionex ASE200

Oven Temperature: 150°C
Pressure: 1750 psi
Static time: 7 minutes (Instrument will automatically perform a 7-minute pre-heat equilibration cycle)
Flush volume: 60% of cell volume
Nitrogen Purge: 180 seconds at 150 psi
Static Cycles: 3

APPENDIX 7

STANDARD OPERATING PROCEDURE (SOP) GEHR3540C

1.0 TITLE General Electric (GE) Hudson River Design Support Sediment Sampling and Analysis Program Standard Operating Procedure for the extraction and cleanup of sediment/solid samples for Polychlorinated Biphenyl (PCB) analysis using the Soxhlet extraction technique by SW-846 Method 3540C for subsequent analysis by SW-846 Method 8082.

(Acknowledgement: This SOP is based substantially on internal method SOPs provided by Northeast Analytical, Inc. of Schenectady, N.Y.)

2.0 PURPOSE The purpose of this SOP is to provide to the chemist the procedures required to perform extractions of PCBs, in sediment/solid sample, using the soxhlet extraction technique and to perform the subsequent extract volume reduction and cleanup for the GE Hudson River Design Support Sediment Sampling and Analysis Program.

3.0 SCOPE The following procedure is utilized by the project laboratories for the extraction and cleanup of PCBs from sediment/solid samples using the soxhlet extraction method for analysis by SW-846 Method 8082.

4.0 COMMENTS The automated solvent extraction may be used in place of the soxhlet extraction at the discretion of the supervising chemist. Time restraints (*i.e.* requested turn around time) may render this method inapplicable, as it requires 18 +/-2 hours of extraction reflux time.

5.0 SAFETY The chemist should have received in-house safety training and should know the location of first aid equipment and the emergency spill/clean-up equipment, before handling any apparatus or equipment. Safety glasses and gloves must be worn when handling glassware and samples.

Polychlorinated biphenyls have been tentatively classified as known or suspected carcinogens. The chemist must review the Material Safety Data Sheets (MSDS) for PCBs and all reagents used in the procedure before handling them. All equipment and solvents should be handled within a lab fume hood.

6.0 REQUIREMENTS The chemist must have an understanding of the methods and requirements of USEPA-SW- 846A "Test Methods for Solid Wastes" Volume 1B: Lab Manual, 3rd edition. Methods 3540C, 3500B, 3620B, 3665A, 3660B. An approved instructor must also certify the chemist to perform the procedure.

7.0 EQUIPMENT

7.1 Water Cooled Condenser: Pyrex 45/50 #3840-MCO or equivalent.

7.2 250mL Round Bottom Flask: Pyrex #4100 or equivalent.

7.3 Soxhlet Repetitive Flushing (reflux) Unit: 45/50 Pyrex #3740-M or equivalent.

7.4 Heating Mantle: Type "VF" laboratory heating mantle #HM0250VF1. (or equivalent)

7.5 Heating Mantle Controller: Glass-Col #PL3122 Minitwin (or equivalent) regulates temperature control of the mantle.

7.6 Analytical Balance: Mettler AG-204 (or equivalent) used to determine sample mass.

7.7 Cellulose Extraction Thimble: Contains sample during soxhlet extraction.

7.8 Sodium Sulfate: Anhydrous (12-60 Mesh), washed with hexane and baked overnight. Used for the laboratory method blank.

7.9 Boiling Chips: Chemware PTFE Boiling Stones P#0919120 (or equivalent)

7.10 Chiller: Pump driven water circulating cooling system cool flow #75 NESLABS Instruments, Inc. (or equivalent)

7.11 Hexane: High Purity Solvent Baxter (Burdick/Jackson) #UN1208 (or equivalent).

7.12 Acetone: High Purity Solvent Baxter (Burdick/Jackson) #UN1090 (or equivalent).

7.13 TurboVap Evaporator: Zymark #ZW640-3 (or equivalent).

7.14 TurboVap Evaporator concentrator tubes: Zymark 250 mL (or equivalent), 0.5 mL endpoint.

7.15 Beakers: Assorted Pyrex: 250 mL, 600 mL, and 1000 mL, used for liquid containment and pipette storage.

7.16 1:1 Hexane/Acetone: 50%/50% by volume solvent mixture prepared in the lab.

7.17 Vials: glass, 8 dram & 4 dram (with Polyseal sealed cap) (20 mL & 10 mL) capacity, for sample extracts.

7.18 Vial Rack: Plastic rack used to hold vials, during all phases of the extract processing.

7.19 Centrifuge: International Equipment Co., Model CL (or equivalent).

7.20 Wrist Shaker: Burrell wrist action shaker, Model 75 and 88 (or equivalent).

7.21 Florisil®: 10% deactivated.

7.22 TBA Reagent: Tetrabutylammonium Hydrogen-Sulfite Reagent (prepared in the laboratory).

7.23 Mercury: Triple distilled Mercury Waste Solutions, Inc, (or equivalent).

7.24 Sulfuric Acid: H_2SO_4 (concentrated) Mallinkrodt #2468 #UN1830 (or equivalent).

7.25 Pipettes: S/P Disposable Serological Borosilicate Pipettes.

1. 1 mL × 1/10
2. 5 mL × 1/10
3. 10 mL × 1/10

Fisher Pasteur Borosilicate glass pipette 9" #72050 (or equivalent)

7.26 4 oz. Jars: Industrial Glassware

8.0 PROCEDURES

8.1 Sample Preparation

8.1.1 Throughout the entire process it should be noted that if the chemist encounters any problems or difficulties with any samples or steps involved, these problems should be brought to the attention of the supervisor and/or quality assurance manager for guidance to proceed and then documented in the extraction logbook.

8.1.2 If the sample is sediment and contains a water layer, decant and discard the layer as aqueous PCB waste. Mix the sample thoroughly and discard any foreign objects such as sticks, rocks or leaves. **Note:** however that the sample may be composed entirely of rock, concrete or some other solid material in which case the entire sample is treated as the solid.

8.2 Sample Extraction

8.2.1 Fill a Pyrex pan with ice cubes and cold water about 1/2 inch deep. As the samples are weighed out, place the beakers or 4-oz. jars in the Pyrex pan to chill for at least 15 minutes prior to the drying step.

8.2.2 Rinse all extraction thimbles with hexane to remove extraneous material. Place thimble into a 100-mL beaker and allow to dry.

8.2.3 Set up one 250-mL glass beaker or 4-oz. jar for each sample. Using the first sample, label a beaker with the sample number, and tare the beaker. Using a metal spatula, add 10 g to 11 g of the wet sample to the beaker. Samples that are observed to be very wet will require additional mass of sample such that the project sensitivity requirements are met. The moisture content of the sample as determined in Section 8.2.4 should be evaluated so that a larger wet-weight sample can be obtained to provide a dry amount of solids to meet the project sensitivity requirements. The amount taken must consider the size limitations of the Soxhlet thimble. The laboratory should target a wet-weight amount of 15 g for very wet samples. Record the weight in the PCB

solid extraction logbook to the nearest tenth of a gram. At this point, a sample for percent total solids may also be taken (see 8.2.4). Place the beaker in the ice bath to chill.

8.2.4 The PCB concentration is to be determined on a dry weight basis and therefore, the percent total solid must be determined. Weigh approximately 5 grams of the previously homogenized sample in a previously weighed, aluminum weighing pan. Record the weight of the pan and the weight of the (pan and sample) in the percent total solids log. Place the sample in a drying oven at 100 to 110°C for at least 8 hours. Record the time placed in the oven and the oven temperature in the percent total solids log. Remove the samples from the drying oven and allow to cool in a desiccator. Weigh the pan and sample.

Calculate the percent solids by:

$$\frac{\{(\text{wt. of pan + dried sample}) - (\text{wt.of pan})\}}{(\text{wt. of wet sample})} \times 100\%$$

NOTE: ALL SAMPLE CONTAINERS ARE TO BE RETURNED TO THE APPROPRIATE REFRIDGERATOR. FOR ALL EMPTY SAMPLE CONTAINERS, SEE THE LABORATORY'S INTERNAL CHEMICAL HYGIENE PLAN FOR PROPER DISPOSAL.

8.2.5 After the sample has been sufficiently chilled, add approximately 10 g of a 1:1 mix of Magnesium Sulfate/Sodium Sulfate to the sample and mix well with a metal spatula. If the sample has not dried after a few minutes, another 10 g may be added. Once the sample is well-dried and free flowing, transfer the sample to a pre-rinsed extraction thimble. Repeat with remaining samples. Set empty mixing beaker and stirring utensil aside for later rinsing into soxhlet extractor to complete sample transfer. Be careful not to add too much drying agent to the sample, if too much is added, the sample may not fit completely in the thimble. In this case the sample will have to be split into two different soxhlets.

8.2.6 Add 200 mL of a 1:1 mixture of hexane/acetone to a 250-mL round bottom flask. Add several boiling chips. Place a soxhlet extractor on top of the round bottom flask. Label the round bottom with the sample number. Place the corresponding thimble into the soxhlet extractor. Rinse corresponding beaker & metal spatula with a few pipettes of hexane. Transfer into thimble. Repeat this step twice more with the same sample, and then repeat all preceding steps with remaining samples. After all samples have been processed, add the specified surrogate and matrix spikes required directly into thimble. The final extract volume concentrations of the surrogate compounds tetrachloro-*meta*-xylene (TCMX) and decachlorobiphenyl (DCB) should be 10 ng/mL and 100 ng/mL, respectively. At this time, the GE Hudson River Design Support Sediment Sampling and Analysis Program does not require the preparation and analysis of matrix spike and/or matrix spike duplicate samples. If requested in the future, the final extract volume

concentration for the spiked Aroclor (Aroclor-1242) in the matrix spike and matrix spike duplicate sample should be 20,000 ng/mL (**Note:** this spike concentration will require a sample dilution to be performed). The final extract volume concentration of the laboratory control sample (LCS) should be 500 ng/mL.

8.2.7 Rinse the inside and the outside connecting joints of the condenser units that will be used to condense the extraction solvent during the soxhlet extraction of the sample. Turn on chiller to cool the condensers.

8.2.8 Place the round bottom flask with attached soxhlet extractor onto a heating mantle and attach condenser unit. Turn corresponding thermostats on to a temperature that will achieve 4-6 solvent cycles per hour. At this time double check soxhlets for any cracks or chips which may leak solvent. Once the solvent begins to boil, a flushing action of once every two to three minutes should be achieved.

8.2.9 The samples should be extracted overnight for a minimum of 16 hours. Once the sample has finished extracting (usually in the morning), turn the heating mantle off and allow samples to cool to room temperature. Turn off the chiller and once cool, rinse the inside of the condenser with several pipette volumes of hexane. Disengage the soxhlet and condenser unit and rinse the joint off as well into the soxhlet.

8.2.10 Move soxhlet units into a chemical fume hood and flush the remaining solvent from the soxhlet extractor by tipping the soxhlet. Using a pair of

long-handled tweezers, pull the thimbles out of the soxhlets one at a time and allow them to drip dry by balancing the thimbles on the tops of the soxhlets. Once dry; remove the thimbles to a Pyrex drying pan for total solvent evaporation.

8.2.11 Rinse the soxhlet with several pipettefull of hexane and tip again to drain into the round bottom. Set the soxhlet aside at this time. Procure the same number of TurboTubes as there are samples. Using an individual TurboTube stand, label a TurboTube with the corresponding sample ID number and place in the holder. Pour the contents of the round bottom into the TurboTube, using a pipette and Hexane to rinse the last drops out of the mouth of the round bottom. Rinse the round bottom with several pipettefull of hexane, swirl gently, and decant into same TurboTube. Repeat this step twice for same sample then repeat all preceding steps for all other samples.

8.2.12 All glassware must be rinsed with technical grade (tech)-Acetone or a "for rinsing-only" labeled solvent, and dried in the hood before other cleaning steps.

8.3 Solvent Reduction: TurboVap Evaporator System

8.3.1 The TurboVap evaporator system is used in place of the Kuderna Danish (KD)-concentrator apparatus. The TurboVap uses a heated water bath and positive pressure nitrogen flow/vortex action. The unit maintains a slight equilibrium imbalance between the liquid and gaseous phase of the solvent

extract, which allows fractional reduction of the solvents without loss of higher boiling point analytes.

8.3.2 Turn the unit on and allow to heat up to the specified temperature for individual solvent use.

8.3.3 As a precaution the TurboVap system regulators should be checked to assure that no residual gas pressure remains within the system and that gas pressure regulators is off before placing samples in the apparatus. Residual gas pressure may cause splashing and cross contamination of samples. To bleed the system of residual gas pressure place an empty TurboTube into the water bath and close the lid. Make sure that the nitrogen gas pressure regulator is turned off. Bleed any residual gas until the regulator output pressure gauge reads "0" psi. Proceed to 8.3.4. Make sure to wipe down all surfaces with Hexane before concentrating samples.

8.3.4 Place the TurboTube containing the samples into the TurboVap and close the lid. Turn on the gas cylinder valve first and then begin slowly turning the pressure regulator on. Keep the gas pressure very low, until the solvent level is decreased, to avoid splashing. Increase the gas pressure as the sample reduces maintaining uniform flow throughout the reduction.

8.3.5 The process for solvent (Hexane/Acetone) reduction takes approximately 20-30 minutes. Do not leave the unit unattended as extracts may be blown

to dryness and PCB loss may occur. Immediately notify a supervisor if an extract is blown to dryness.

8.3.6 Concentrate the solvent to approximately 5.0 mL. Remove the samples from the TurboVap and place in the rack. **Note:** Not all samples will evaporate at the same rate; sample extracts containing large amounts of petroleum or other non-volatile liquids may stop reducing before the 5.0 mL point is achieved. Samples, which stop reducing, should be removed as soon as possible.

8.3.7 Quantitatively transfer the sample extract with a Pasteur pipette into an appropriate volumetric flask (25 mL for soil extracts). Rinse the TurboTube with 3 Pasteur pipettes of hexane, and then transfer the hexane rinse to the volumetric. Repeat the hexane rinse two more times for a total of three Hexane rinses of the TurboTube. After the sample has been transferred, rinse the Pasteur pipette with 0.5 mL of hexane into the volumetric flask. Add hexane to the volumetric meniscus mark. Invert the volumetric flask at least three times to mix completely. Decant the contents into a pre-labeled 8-dram vial.

8.3.8 All dirty glassware must be rinsed with tech-Acetone or a "For Rinsing-Only" labeled solvent and dried in the fume hood before being washed.

8.4 Sample Extract Cleanup

Most extracts of environmental samples that are to be analyzed for PCBs by gas chromatography with electron capture detection contain co-extracted xenobiotics and other interfering substances which must be removed before accurate chromatographic analysis can be performed.

Sulfuric acid, sulfur removal and Florisil® clean-ups should be performed on every sample. The sample preparation chemist in the extraction logbook records the sequence and number of repeats of cleanup steps performed.

Sample extract cleanups are performed on set volume extracts. The set volume is 25 mL for sediment/solid samples.

8.4.1 Sulfuric Acid Wash

8.4.2 The concentrated sulfuric acid treatment removes hydrocarbons and other organic compounds that are co-extracted with the PCB residues.

8.4.3 Chill the sample to approximately 0°C. Add 5.0 mL of concentrated H_2S0_4 and shake for 30 seconds by hand, centrifuge for approximately 1 minute, transfer approximately 20 mLs of the Hexane upper layer to an 8-dram vial.

8.4.4 Repeat 8.4.2 if the sample extract appears to be heavily loaded (opaque) with colored material. Two to three acid washes may be required. **Note:** it is entirely possible that all colored material will not be removed from the extract.

8.5 Elemental Sulfur Clean-up

8.5.1 Elemental sulfur is soluble in the extract solvents used for sediment and soil samples. It is commonly found sediment/soil samples, decaying organic material and some industrial wastes. Large amounts of sulfur can cause the electron capture detector (ECD) to signal saturate for long periods during the elution envelope of PCBs. Even small amounts of sulfur can interfere with PCB measurement as a co-eluting chromatographic peak.

8.5.2 Two techniques exist for the elimination of elemental sulfur in PCB extracts. Mercuric precipitation (Mercury Shake) and the Tetrabutylammonium (TBA) sulfite procedure. Tetrabutylammonium sulfite causes the least amount of degradation of a broad range of pesticides and organics compounds, while mercury may degrade organophosphorus and some organochlorine pesticides. The TBA procedure also has a higher capacity for samples containing high concentrations of elemental sulfur.

8.6 Removal of Sulfur Using Mercury

Note: Mercury is a highly toxic metal. All operations involving mercury should be performed within a hood. Prior to using mercury, the chemist should become acquainted with proper handling and emergency spill/clean-up procedures associated with this metal and must have reviewed the material safety data sheet MSDS.

8.6.1 Add 1-3 drops of mercury to the sample extracts, cap, and place on the wrist shaker for 30 minutes. The sulfur is converted to mercuric sulfide and precipitates out of the sample extract. A black precipitate may be seen in sample extracts containing elemental sulfur.

8.6.2 Remove the sample extracts from the wrist shaker and place in the centrifuge at a setting and duration appropriate to spin down the solids.

8.6.3 Transfer the sample extract to a new 8-dram vial.

8.6.4 The precipitated sulfur can be removed from the extract by performing a sulfuric acid clean-up or Florisil® slurry (discussed in 8.8.0).

8.7 Removal of Sulfur using TBA Sulfite

8.7.1 The TBA procedure removes elemental sulfur by conversion to the thiosulfate ion, which is water-soluble.

8.7.2 Add 2.0 mL TBA Sulfite Reagent, 1.0 mL 2-propanol, and approximately 0.65 g of sodium sulfite crystals to the extract and shake for at least 5 minutes on the wrist shaker and observe. An excess of sodium sulfite must remain in the sample extract during the procedure. If the sodium sulfite crystals are entirely consumed add one or two more aliquots (approximately 0.65 g) to extract and observe.

8.7.3 Place the samples on the wrist shaker for 45 minutes observing at 15-minute intervals to make sure that the sodium sulfite is not consumed. Add 5 mL organic free water and shake for ten minutes.

8.7.4 Place the samples into the centrifuge at a setting and duration appropriate to spin down the solids.

8.7.5 Transfer the hexane layer to a new 8-dram vial and cap.

8.8 Florisil® Adsorption (Slurry)

8.8.1 The Florisil® slurry removes co-extracted polar compounds, residual water, and residual acid and is recommended as the final cleanup step before the extract is submitted for GC analysis.

8.8.2 Add approximately 3 grams of tested and approved 10% deactivated Florisil® to each vial containing the sample extract.

8.8.3 Vigorously shake the vial for approximately 1 minute by hand or on the wrist shaker.

8.8.4 Place the vial(s) into the centrifugeat a setting and duration appropriate to spin down the solids.

8.8.5 Transfer the extract to a clean 8-dram vial.

8.9 Extract Screening and Dilution:

8.9.1 Screening PCB extracts by GC to determine the approximate concentration before final analysis is highly recommended. If possible, prior site history and estimates of sample concentration will be provided by the field personnel and may be used to determine what, if any, extract dilution is necessary.

8.9.2 The supervising chemist is responsible for determining initial screening dilutions. Extract dilutions are prepared by transferring an aliquot of the original sample extract into a vial containing the correct amount of "make up" volume of hexane. Dilutions must be recorded in the instrument logbook or in the data system.

8.9.3 Perform the dilution using appropriate disposable volumetric pipettes to transfer the extract and to add the make-up volume of hexane. Make sure that the vial is properly labeled. Cap and invert the vial at least three times to thoroughly mix the extract with the solvent.

8.9.4 Transfer 1 mL of the extract to a labeled 1.5-mL GC autosampler vial. Record the screening dilution along with the extract volume, the sample mass, and the percent total solids. Submit the information with the sample extracts to the GC analyst.

9.0 QUALITY CONTROL

9.1 This section outlines the necessary quality control samples that need to be instituted at the time of sample extraction. The data from these quality control samples is maintained to document the quality of the data generated.

9.2 9.2 With each batch of samples to be extracted a method blank is processed. The method blank is carried through all stages of sample preparation steps (including clean-up steps). For sediment/solid samples, a laboratory sodium sulfate blank is processed.

9.3 At this time, the GE Hudson River Design Support Sediment Sampling and Analysis Program does not require the preparation and analysis of matrix spike and/or matrix spike duplicate samples. If requested in the future, a matrix spike for Aroclor-1242 is to be analyzed at a rate of 1 matrix spike per every 20 samples at a concentration of 20,000 ng/mL in the extract (**Note:** this spike concentration will require a sample dilution to be performed). Also a matrix spike duplicate sample is to be analyzed at a rate of 1 per every 20 samples.

9.4 A QC reference check standard (Laboratory Control Sample [LCS]) is also prepared and analyzed for Aroclor-1242 at a concentration of 500 ng/mL in the extract. For sediment/solid samples, sodium sulfate is used.

9.5 Surrogate compounds are added to each sample, matrix spike, matrix spike duplicates, duplicate, method blank, and QC reference check standard LCS at time of extraction. The surrogate compounds TCMX and DCB are to be added for final extract concentrations of 10 ng/mL and 100 ng/mL, respectively.

10.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

10.1 Pollution Prevention: see laboratory's internal SOPs

10.2 Waste Management: see laboratory's internal SOPs

11.0 REFERENCES

1. U.S. EPA SW-846 "Test Methods for Evaluating Solid Waste; Volume 1B Laboratory Manual Physical/Chemical Methods", Office of Solid Waste and Emergency Response, Third Edition, Final Update III, December 1996.

2. "Guide to Environmental Analytical Methods", Third Edition, Genium Publishing Corporation, 1996.

12.0 ATTACHMENTS

1. Method Outline

ATTACHMENT 1
PCBs IN SOIL/SEDIMENT
OUTLINE FOR SOXHLET EXTRACTION

1. PREPARE SAMPLE FOR EXTRACTION
2. RINSE EXTRACTION THIMBLES
3. WEIGH SAMPLE AND RECORD WEIGHT
4. DRY SAMPLES
5. ADD SAMPLE TO THIMBLES
6. SET UP SOXHLET EXTRACTOR APPARATUS
7. ADD SURROGATES, MATRIX SPIKE, AND LABORATORY CONTROL SAMPLE SPIKE
8. EXTRACT SAMPLE FOR APPROXIMATELY 16 HOURS
9. BREAKDOWN SOXHLET EXTRACTOR APPARATUS
10. TRANSFER SOLVENT TO TURBOTUBE
11. SOLVENT REDUCTION, USING THE ZYMARK TURBOVAP EVAPORATION SYSTEM
12. TRANSFER AND SET VOLUME
13. EXTRACT CLEANUP (ACID, MERCURY OR TBA, AND FLORISIL)
14. EXTRACT DILUTION
15. GC SCREENING/ ANALYSIS

APPENDIX 8

STANDARD OPERATING PROCEDURE (SOP) GEHR680

GENERAL ELECTRIC (GE) HUDSON RIVER DESIGN SUPPORT

SEDIMENT SAMPLING AND ANALYSIS PROGRAM

STANDARD OPERATING PROCEDURE FOR THE DETERMINATION OF PCBs IN SEDIMENT BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY BY EPA METHOD 680

Revision No.: 1

July 16, 2002

INDEX

1. SCOPE AND APPLICATION

1.1. This method provides procedures for mass spectrometric determination of polychlorinated biphenyls (PCBs) in sediment for the GE Hudson River Design Support Sediment Sampling and Analysis Program (SSAP). This method is applicable to samples containing PCBs as single congeners. PCBs are identified and measured as isomer groups (*i.e.,* by level of chlorination). The existence of 209 possible PCB congeners makes impractical the listing of the Chemical Abstracts Service Registry Number (CASRN) for each potential method analyte. Because PCBs are identified and measured as isomer groups, the non-specific CASRN for each level of chlorination is used to describe method analytes.

Analyte(s)	Formula	CASRN
PCBs		
Monochlorobiphenyls	$C_{12}H_9Cl$	27323-18-8
Dichlorobiphenyls	$C_{12}H_8Cl_2$	25512-42-9
Trichlorobiphenyls	$C_{12}H_7Cl_3$	25323-68-6
Tetrachlorobiphenyls	$C_{12}H_6Cl_4$	26914-33-0
Pentachlorobiphenyls	$C_{12}H_5Cl_5$	25429-29-2
Hexachlorobiphenyls	$C_{12}H_4Cl_6$	26601-64-9
Heptachlorobiphenyls	$C_{12}H_3Cl_7$	28655-71-2
Octachlorobiphenyls	$C_{12}H_2Cl_8$	31472-83-0
Nonachlorobiphenyls	$C_{12}HCl_9$	53742-07-7
Decachlorobiphenyls	$C_{12}Cl_{10}$	2051-24-3

1.2 A Method Detection Limit (MDL) study will be performed on a representative instrument in accordance with the procedures described in 40 CFR Part 136, Appendix B prior to analysis of sediment samples for the SSAP. A clean sodium sulfate will be used as the matrix for this MDL study. Detection limits vary among method analytes and with sample matrix, sample preparation procedures, condition of the GC/MS system, type of data acquisition, and individual samples. Detection limits for individual PCB congeners increase with increasing number of chlorine atoms, with the detection limit for decachlorobiphenyl being about 2 times higher than that of a monochlorobiphenyl. The detection limit for total PCBs will depend on the number of individual PCB congeners present. SIM data acquisition procedures reduce the detection limit for PCBs by at least a factor of three.

2. SUMMARY OF METHOD

In general, samples are extracted with a pesticide-grade solvent. The extracts are further processed by concentrating or diluting, depending on the PCB concentration, and carried through a series of cleanup techniques. The sample is then analyzed by injecting the extract onto a gas chromatographic system and the PCBs detected by a mass spectrometer.

This SOP provides detailed instructions for gas chromatographic/mass spectrometer conditions, calibration, and analysis of PCBs by gas chromatography/mass spectrometry (GC/MS). Sediment extraction procedures are covered in separate standard operating procedures. It is expected that the extracts generated for analysis by SOP GEHR8082 will be used for analysis by this method (GEHR680) to provide paired total PCB results by both methods for the same extract.

Sample extract components are separated with capillary column gas chromatography (GC) and identified and measured with low resolution, electron ionization mass spectrometry (MS). An interfaced data system (DS) to control data acquisition and to store, retrieve, and manipulate mass spectral data is essential. Selected-ion-monitoring (SIM) data are to be acquired.

A Varian Saturn Ion Trap GC/MS will be used by Northeast Analytical, Inc. (NEA) to perform this analysis. Varian uses a proprietary field-modulated Wave-Board technology to selectively trap only those ions of interest. Background ions are not stored. This allows for a much cleaner spectrum and a considerable increase in sensitivity since the trap's capacity is dedicated to these ions of interest. Additionally, the selected storage mass range (Method 680 requires scanning ions across 5 mass ranges) is time programmable so that many different target analytes can be selectively stored relative to the background matrix. Varian refers to this mode of operation as Selected Ion Scanning (SIS) mode. SIS is Varian's term for Selected Ion Monitoring (SIM) common to most other mass spectrometers. SIS allows the Saturn Ion Trap to store many more masses than traditional SIM techniques without a corresponding loss of sensitivity. The more common term SIM (versus SIS) will be used throughout SOP for ease of reference.

Two surrogate compounds are added to each sample before sample preparation; these compounds are tetrachloro-*meta*-xylene (TCMX) and decachlorobiphenyl (DCB). Two internal standards, chrysene-d_{12} and phenanthrene-d_{10}, are added to each sample extract before GC/MS analysis and are used to calibrate MS response. Each concentration measurement is based on an integrated ion abundance of one characteristic ion.

PCBs are identified and measured as isomer groups or homologs (*i.e.*, by level of chlorination). A concentration is measured for each PCB isomer group total; total PCB concentration in each sample extract is obtained by summing isomer group concentrations.

Nine selected PCB congeners are used as calibration standards, and one internal standard, chrysene-$d_{12,}$ is used to calibrate MS response to PCBs, unless sample conditions require the use of the second internal standard, phenanthrene-d_{10}.

3. DEFINITIONS

3.1 CONCENTRATION CALIBRATION SOLUTION (CAL) -- A solution of method analytes used to calibrate the mass spectrometer response.

3.2 CONGENER NUMBER -- Throughout this method, individual PCBs are described with the number assigned by Ballschmiter and Zell (2). (This number is also used to describe PCB congeners in catalogs produced by Ultra Scientific, Hope, RI.)

3.3 INTERNAL STANDARD -- A pure compound added to a sample extract in known amounts and used to calibrate concentration measurements of other compounds that are sample components. The internal standard must be a compound that is not a sample component.

3.4 LABORATORY PERFORMANCE CHECK SOLUTION (LPC) -- A solution of method analytes, surrogate compounds, and internal standards used to evaluate the performance of the GC/MS with respect to a defined set of method criteria.

3.5 METHOD BLANK -- An aliquot of reagent water or neutral solid reference material that is treated as a sample. It is exposed to all glassware and apparatus, and all method solvents, reagents, internal standards, and surrogate compounds are used. The extract is concentrated to the final volume used for samples and is analyzed the same as a sample extract.

3.6 LABORATORY SPIKE DUPLICATE SAMPLE-- One aliquot (LSD) of a sample is analyzed before fortification with any method analytes. In the laboratory, a known quantity of method analytes (LSA) is added to two independent aliquots of the same sample, and final analyte concentrations (LF1 and LF2) are measured with the same analytical procedures used to measure LSD. These analyses are more commonly referred to as matrix spike (MS) and matrix spike duplicate (MSD) samples. MS/MSD analyses are not required by US EPA for the GE Hudson River SSAP.

3.7 LABORATORY SURROGATE SPIKE

3.7.1 Measured Value (LS1) -- Surrogate compound concentration measured with the same procedures used to measure sample components.

3.7.2 Theoretical Value (LS2) -- The concentration of surrogate compound added to a sample aliquot before extraction. This value is determined from standard gravimetric and volumetric techniques used during sample fortification.

3.8 METHOD DETECTION LIMIT (MDL) -- A statistically determined value (1) indicating the minimum concentration of an analyte that can be identified and measured in a sample matrix with 99% confidence that the analyte concentration is greater than zero. This value varies with the precision of the replicate measurements used for the calculation.

3.9 PERFORMANCE EVALUATION SAMPLE -- A sample containing known concentrations of method analytes that has been analyzed by multiple laboratories to determine statistically the accuracy and precision that can be expected when a method is performed by a competent analyst. Analyte concentrations are unknown to the analyst.

3.10 QUALITY CONTROL (QC) CHECK OR LABORATORY CONTROL SAMPLE (LCS) -- A sample containing known concentrations of analytes that is analyzed by a laboratory to demonstrate that it can obtain acceptable identifications and measurements with procedures to be used to analyze environmental samples containing the same or similar analytes. Analyte concentrations are known by the analyst.

3.11 SURROGATE COMPOUND -- A compound not expected to be found in the sample is added to a sample aliquot before extraction and is measured with the same procedures used to measure sample components. Associated with the surrogate compound are two values, laboratory surrogate spike - measured value (LS1) and laboratory surrogate spike - theoretical value (LS2). The purpose of a surrogate compound is to monitor method performance with each sample.

4. INTERFERENCES

4.1 Interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing equipment. Method blanks are analyzed routinely to demonstrate that these materials are free of interferences under the analytical conditions used for samples.

4.2 To minimize interferences, glassware (including sample bottles) should be meticulously cleaned. As soon as possible after use, rinse glassware with the last solvent used. Than wash with detergent in hot water and rinse with tap water followed by distilled water. Drain dry. Heating in a muffle furnace at 450°C for a few hours may be used as a further cleaning technique, but does not have to be performed provided that method blanks demonstrate glassware cleanliness. After cooling, store glassware inverted or covered with aluminum foil. Before using, rinse each piece with an appropriate solvent. Volumetric glassware should not be heated in a muffle furnace.

4.3 For PCBs, interference can be caused by the presence of much greater quantities of other sample components that overload the capillary column; additional sample extract preparation procedures must then be used to eliminate interferences (refer to the applicable extraction SOPs for extract cleanup procedures). Capillary column GC retention time and the compound-specific characteristics of mass spectra eliminate many interferences that formerly were of concern with PCB determinations with electron capture detection. The approach and identification criteria used in this method for PCBs eliminate interference by most chlorinated compounds other than other PCBs. With the isomer group approach, coeluting PCBs that contain the same number of chlorines are identified and measured together. Therefore, coeluting PCBs are a problem only if they contain a different number of chlorine atoms. This interference problem is obviated by rigorous application of the identification criteria described in this method.

5. SAFETY

5.1 Safety glasses and disposable gloves must be worn when handling samples and extracts.

5.2 All manipulations of sample extracts should be conducted inside a chemical fume hood. The analyst should minimize manipulation of sample extracts outside of a fume hood.

5.3 Safe laboratory practices should be followed by the analyst at all times when conducting work in the lab. The analyst should refer to the reference file of material safety data sheets to familiarize themselves with the precautions of handling applicable solvents and chemicals used to process samples. The analyst should refer to the project laboratory's internal chemical hygiene plan for further safety information.

5.4 Samples remaining after analysis should be disposed of through the project laboratory's internal disposal plan. Refer to the project laboratory's internal standard operating procedures for disposal of laboratory waste.

6. APPARATUS AND EQUIPMENT

6.1 COMPUTERIZED GC/MS

The specific GC and MS operating parameter to be used are summarized on Table 1.

6.1.1 The GC must be capable of temperature programming and be equipped with all required accessories, such as syringes, gases, and a capillary column. The GC injection port must be designed for capillary columns. Splitting injections is not recommended.

6.1.2 SIM mass spectral data are obtained with electron ionization at a nominal electron energy of 70 eV. To ensure sufficient precision of mass spectral data, the required MS scan rate must allow acquisition of at least five full-range mass spectra or five data points for each monitored ion while a sample component elutes from the GC. The MS must produce a mass spectrum meeting all criteria for $\leq$ 20 ng of decafluorotriphenylphosphine (DFTPP) introduced through the GC inlet.

6.1.3 An interfaced data system (DS) is required to acquire, store, reduce, and output mass spectral data. The DS must be capable of searching a data file for specific ions and plotting ion abundances versus time or spectrum number to produce selected ion current profiles (SICPs) and extracted ion current profiles (EICPs). Also required is the capability to obtain chromatographic peak areas between specified times or spectrum numbers in SICPs or EICPs. Total data acquisition time per cycle should be $\geq$ 0.5 s and must not exceed 1.5 s.

6.1.4 SIM – For SIM data acquisition, the DS must be equipped with software capable of acquiring data for multiple groups of ions, and the DS must allow automated and rapid changes of the set of ions being monitored. The SIM program must be capable of acquiring data for five groups (or mass ranges) each consisting of $\leq$ 27 ions each. The times spent monitoring ions during sample analyses must be the same as the times used when calibration solutions were analyzed.

6.2 GC COLUMN – A 30 m $\times$ 0.25 mm ID fused silica capillary column coated with a 0.25 μm film, Durabond-XLB, Agilent Technologies is required. Operating conditions known to produce acceptable results with this column are shown in Table 1. Separation of PCB calibration congeners with a DB-XLB column and those operating conditions is shown in Figure 1. Figure 2 shows a chromatogram of the PCB Window Defining Mixture used to determine retention time windows for the five ion groups for SIM data acquisitions.

6.3 MISCELLANEOUS EQUIPMENT

6.3.1 Volumetric flasks -- 2-mL, 5-mL, 10-mL, 25-mL, and 50-mL with ground glass stoppers.

6.3.2 Microsyringes -- various standard sizes 9.

6.3.3 Analytical Balance -- capable of accurately weighing to 0.0001 g.

7. REAGENTS AND CONSUMABLE MATERIALS

7.1 SOLVENTS -- High purity, distilled-in-glass hexane and methylene chloride. For precise injections with splitless injectors and capillary columns, all samples and standards should be contained in the same solvent. Effects of minor variations in solvent composition (*i.e.,* small percentage of another solvent remaining in hexane extracts) are minimized with the use of internal standards. (External standard calibration is not acceptable.)

7.2 SODIUM SULFATE - ACS, granular, anhydrous. Purify by heating at 400°C for 4 h in a shallow tray.

7.3 MS PERFORMANCE CHECK SOLUTION - Prepare a 10 ng/μL solution of decafluorotriphenylphosphine (DFTPP) in an appropriate solvent.

7.4 INTERNAL STANDARDS - Chrysene-d_{12} and phenanthrene-d_{10} are used an internal standards. They are added to each sample extract just before analysis and are contained in all calibration/performance check solutions and quality control samples.

7.5 SURROGATE COMPOUNDS – TCMX and DCB are added to every sample before extraction and are included in every calibration/performance check solution and quality control samples.

7.6 PCB CONCENTRATION CALIBRATION CONGENERS - The nine individual PCB congeners listed on Table 2 are used as concentration calibration compounds for PCB determinations. One isomer at each level of chlorination is used as the concentration calibration standard for all other isomers at that level of chlorination, except that decachlorobiphenyl (Cl_{10}) is used for both Cl_9 and Cl_{10} isomer groups. The basis for selection of these calibration congeners has been reported (6).

7.7 PCB RETENTION TIME CONGENERS FOR SIM DATA ACQUISITION -- Knowledge of the retention time of certain congeners is necessary to determine when to acquire data with each ion set. Two concentration calibration congeners also serve as retention time congeners; the first eluting Cl_1-PCB indicates the time when data acquisition must have been initiated for ion set #1, and the Cl_{10}-PCB indicates when all PCBs have eluted. A PCB Window Defining Mixture Standard (AccuStandard, Inc., catalog item C-WDM or equivalent) is analyzed at a concentration of 2.5ug/mL for each PCB congener. The PCB Window Defining Mixture Standard contains the first and last eluting PCB congener for each Homolog group. The following four congeners are used from this standard to define the five retention time segments for the five Ion Set Groups: BZ#104, BZ#77, BZ#202, and BZ#189. (See Sect. 9.2.3.1.3 for Ion Set Segments).

7.8 PCB SOLUTIONS

7.8.1 Stock Solutions of PCB Calibration Congeners -- Prepare a stock solution of each of the nine PCB concentration calibration congeners at a concentration of 1 μg/μL in hexane. Place each solution in a clean glass vial with a Teflon-lined screw cap and store at 4°C if solutions are not to be used right away. Solutions are stable indefinitely if solvent evaporation is prevented. CAUTION: Each time a vial containing small volumes of solutions is warmed to room temperature and opened, a small volume of solvent in the vial headspace evaporates, significantly affecting concentration. Solutions should be stored with the smallest possible volume of headspace, and opening vials should be minimized.

7.8.2 PCB Primary Dilution Standard -- Take aliquots of the stock solutions of the nine PCB concentration calibration congeners and mix together-in the proportions of one part of each solution of the Cl_1 (#1), Cl_2 (#5), and Cl_3 (#29) congeners, two parts of each solution of the Cl_4 (#50), Cl_5 (#87), and Cl_6 (#154) congeners, three parts of each solution of the Cl_7 (#188) and Cl_8 (#200) congeners, and five parts of the Cl_{10} (#209) congener solution. This will provide a primary dilution standard solution of the composition shown on Table 3. Calculate the concentration in μg/μL; use three significant figures. Place each solution in a clean glass vial with a Teflon-lined screw cap and store at 4°C. Mark the meniscus on the vial wall to monitor solution volume during storage; solutions are stable indefinitely if solvent evaporation in prevented.

7.9 INTERNAL STANDARD (IS) SOLUTION

7.9.1 IS solution (for SIM CALs) – Phenanthrene-d_{10} and chrysene-d_{12} at a concentration of 40 ng/μL (ppm). A stock standard is prepared by transferring 1 mL of 1000 ng/μL phenanthrene-d_{10} and 0.5 mL of 2000 ng/μL chrysene-d_{12} to 25 mL hexane to provide a 40 ng/μL (ppm) solution.

7.10 CAL FOR SIM DATA ACQUISITION -- One set of six solutions is needed for PCB determinations. Appropriate concentrations of SIM CALs are given on Table 4a and 4b. Solutions are prepared by diluting appropriate primary dilution standards and adding an appropriate volume of IS solution #2. Four (4) μL of IS solution (7.9.1) will be added to 200 μL of extract to provide phenanthrene-d_{10} and chrysene-d_{12} at a concentration of 0.80 μg/μL (ppm) in the extract. The CAL6 level is prepared by using 600 μL of primary dilution standard plus 400 μL of Hexane. This gives a concentration for decachlorobiphenyl of 15.0 ug/mL. The GC/MS calibration standard is prepared by taking 200 μL of this standard and spiking with four (4) μL of internal standard. Only decachlorobiphenyl will be calibrated from this sixth standard, leaving all analytes as a five-point calibration.

7.11 Calculate the concentration (two significant figures if <100 and three significant figures if >100 ng/μL) of each component in each solution. Note: Concentrations presented in tables are only approximate.

7.12 LABORATORY PERFORMANCE CHECK SOLUTION - The Medium CAL is used as the laboratory performance check solution (LPC) to verify response factors and to demonstrate adequate GC resolution and MS performance.

7.13 PCB Window Defining Mixture Standard – This standard is used as purchased at 2.5ug/mL per congener. It is analyzed by full scan to provide a check on retention time for the four PCB congeners used to established retention time segments for SIM data acquisition.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

Sample collection, preservation, and storage of sediment samples is addressed in the Design Support Sediment Sampling Analysis Program Field Sampling Plan (FSP) and Quality Assurance Project Plan (QAPP).

9. CALIBRATION

Demonstration and documentation of acceptable initial calibration is required before any samples are analyzed and is required intermittently throughout sample analyses as dictated by results of continuing calibration checks. After initial calibration is successfully performed, a continuing calibration check is required at the beginning and end of each 12-h period during which analyses are performed.

9.1 DATA ACQUISITION -- SIM data acquisition is to be used.

9.1.1 SIM data acquisition will provide an increase in sensitivity from full-range data acquisition by at least a factor of three for PCB determinations.

9.2. INITIAL CALIBRATION

9.2.1 Calibrate and tune the MS with standards and procedures prescribed by the manufacturer with any necessary modifications to meet requirements defined in this SOP.

9.2.2 Inject a 2-μL aliquot of the 10 ng/μL DFTPP solution and acquire a mass spectrum that includes data for m/z 45-450. If the spectrum does not meet all criteria (Table 5), the MS must be hardware tuned to most all criteria before proceeding with calibration.

9.2.3 SIM Calibration – Inject a 2-μL aliquot of the Medium CAL. Acquire at least five data points for each ion during elution of each GC peak. Total cycle time should be ≥ 0.5 s and <1.5 s. CAUTION: When acquiring SIM data, GC operating conditions must be carefully reproduced for each analysis to provide reproducible retention times; if not, ions will not be monitored at the appropriate times.

9.2.3.1 SIM Calibration for PCB determinations

9.2.3.1.1 Data will be acquired with the five ion sets (≤ 27 ions each) shown on Tables 6a and 6b.

9.2.3.1.2 The time (scan number) for initiation of data acquisition with each ion set must be carefully determined from the retention times (scan numbers) of the retention time congeners. Approximate relative retention times of calibration congeners and approximate relative retention time windows for PCB isomer groups are shown on Table 7. (Also see Figure 1 and Figure 2.)

9.2.3.1.3 SIM data acquisition with five ion sets. Begin data acquisition with Ion Set #1 before elution of PCB congener #1, the first eluting Cl_1-PCB. Stop acquisition with Ion Set #1 and begin acquisition with Ion Set #2 just (approximately 10 seconds) before elution of PCB congener #104, the first eluting Cl_5-PCB. Stop acquisition with Ion Set #2 and begin acquisition with Ion Set #3 just (approximately 10 s) after elution of PCB congener #77, the last eluting Cl_4-PCB. Stop acquisition with Ion Set #3 and begin acquisition with ion Set #4 just (approximately 10 s) before elution of PCB congener #202, the first eluting Cl_8-PCB. Stop acquisition with Ion Set #4 and begin acquisition with Ion Set #5 just (approximately 10 s) after elution of PCB congener #189, the last eluting Cl_7-PCB, stop acquisition of Ion Set #5 after Cl_{10}-PCB elution.

9.2.4 Performance Criteria

9.2.4.1 SIM PCB Data

9.2.4.1.1 GC separation -- Baseline separation of PCB congener #87 from congeners #154 and #77, which may coelute.

9.2.4.1.2 MS sensitivity -- Signal/noise ratio of $\geq$ 5 for m/z 499 of PCB congener #209, Cl_{10}-PCB, and for m/z 241 of chrysene-d_{12}.

9.2.4.1.3 MS calibration -- Abundance of $\geq$ 70% and $\leq$ 95% of m/z 500 relative to m/z 498 for congener #209, Cl_{10}-PCB.

9.2.5 Replicate Analyses of CALs -- If all performance criteria are met, analyze one 2-uL aliquot of each of the other four CALs.

9.2.6 Response Factor Calculation

9.2.6.1 Calculate five response factors (RFs) for each PCB calibration congener and surrogate compound relative to both ISs (see Sect. 12.4.2), phenanthrene-d_{10} and chrysene-d_{12}.

$$RF = A_x Q_{is} / A_{is} Q_x$$

where:

A_x = integrated ion abundance of quantitation ion for a PCB calibration congener or a surrogate compound,

A_{is} = integrated ion abundance of m/z 240, the quantitation ion when chrysene-d_{12} is used as the internal standard or m/z 188, the quantitation ion when phenanthrene-d_{10} is used as the internal standard,

Q_{is} = injected quantity of chrysene-d_{12} or phenanthrene-d_{10},

Q_x = injected quantity of PCB calibration congener or surrogate compound.

RF is a unitless number, units used to express quantities must be equivalent.

9.2.7 Response Factor Reproducibility -- For each PCB calibration congener and surrogate compound, calculate the mean RF from analyses of each of the five CALs. When the RSD exceeds 20%, analyze additional aliquots of appropriate CALs to obtain an acceptable RSD of RFs over

the entire concentration range, or take action to improve GC/MS performance.

9.2.8 SIM Analyte Retention Time Reproducibility

9.2.8.1 PCB determinations - Absolute retention times of PCB congeners #77, #104, #202, and #189 should not vary by more than ±10 s from one analysis to the next. (Retention time reproducibility is not as critical for congeners #1 and #209 as for the other four congeners, which are used to determine when ion sets are changed.)

9.2.9 Record a spectrum of each CAL component.

9.3 CONTINUING CALIBRATION CHECK

9.3.1 With the following procedures, verify initial calibration at the beginning and end of each 12-h period during which analyses are to be performed.

9.3.2 Calibrate and tune the MS with standards and procedures prescribed by the manufacturer.

9.3.3 Analyze a 2-μL aliquot of the DFTPP solution and ensure acceptable MS calibration and performance (Table 5).

9.3.4 Inject a 2-μL aliquot of the Medium CAL and analyze with the same conditions used during Initial Calibration.

9.3.5 Demonstrate acceptable performance for criteria described in Sect. 9.2.4.

9.3.6 Determine that neither the area measured for m/z 240 for chrysene-d_{12} nor that for m/z 188 for phenanthrene-d_{10} has decreased by more than 30% from the area measured in the most recent previous analysis of a calibration solution or by more than 50% from the mean area measured during initial calibration.

9.3.7 Response Factor Reproducibility -- For an acceptable Continuing Calibration Check, the measured RF for each analyte/surrogate compound must be within ±20% of the mean value calculated (Sect. 9.2.6) during Initial Calibration. If not, remedial action must be taken; recalibration may be necessary.

9.3.8 SIM Analyte Retention Time Reproducibility -- Demonstrate and document acceptable (Sect. 9.2.8) reproducibility of absolute retention

times of appropriate PCB retention time congeners by analysis of the PCB Window defining mixture in full scan mode.

9.3.9 Remedial actions must be taken if criteria are not met; possible remedies are:

9.3.9.1 Check and adjust GC and/or MS operating conditions.

9.3.9.2 Clean or replace injector liner.

9.3.9.3 Flush column with solvent according to manufacturers instructions.

9.3.9.4 Break off a short portion (approximately 0.33 m) of the column; check column performance by analysis of performance check solution.

9.3.9.5 Replace GC column; performance of all initial calibration procedures is then required.

9.3.9.6 Adjust MS for greater or lesser resolution.

9.3.9.7 Calibrate MS mass scale.

9.3.9.8 Prepare and analyze new concentration calibration/performance check solution.

9.3.9.9 Prepare new concentration calibration curve(s).

10. QUALITY CONTROL

The QC sample extracts (method blank and laboratory control sample) associated with the sediment sample extracts will be performed at a frequency of one method blank and one laboratory control sample (LCS) per 20 sample extracts. As many field sediment sample extracts originating from multiple laboratories and analysis extraction batches will be selected for Method 680 analysis, a representative method blank and LCS extract that has passed GEHR8082 acceptance criteria will be selected to be run with up to 20 sediment sample extracts for this analysis. Sediment sample extracts for Method 680 analysis will be selected from SOP GEHR8082 analysis batches where the method blank and LCS passed SOP GEHR8082 criteria. If this is not always possible, then the method and LCS that failed SOP GEHR8082 criteria will also be run by Method 680 (SOP GEHR680).

10.1 Method Blank – The extracts for this analysis will be the same extracts as those generated for the analysis of total PCBs as Aroclors by SOP GEHR8082.

10.1.1 A method blank must contain the same amount of surrogate compounds and internal standards that is added to each sample.

10.1.2 Analyze a method blank before any samples are analyzed.

10.1.3 An acceptable method blank contains no method analyte at a concentration greater than its reporting limit (RL) for the PCB homologue and contains no additional compounds with elution characteristics and mass spectral features that would interfere with identification and measurement of a method analyte at its RL. If the method blank that was extracted along with a batch of samples is contaminated, the entire batch of samples must be reanalyzed.

10.1.4 Corrective action for unacceptable method blank -- Check solvents, reagents, apparatus and glassware to locate and eliminate the source of contamination before any samples are extracted and analyzed. Purify or discard contaminated reagents and solvents.

10.2 CALIBRATION - Included among initial and continuing calibration procedures are numerous quality control checks to ensure that valid data are acquired (see Sect. 9). Continuing calibration checks are accomplished with results from analysis of the medium-level calibration solution and the PCB Window Defining Mixture to monitor criteria times for the five (5) ion sets.

10.2.1 If some criteria are not met for a Continuing Calibration Check after a 12-h period during which samples were analyzed, those samples must be reanalyzed. Those criteria are: GC performance (Sect. 9.2.4), MS calibration as indicated by DFTPP spectrum (Sect. 9.2.2), and MS sensitivity as indicated by area of internal standards (Sect. 9.3.6).

10.2.2 When other criteria in Sect. 9.2 are not met, results for affected analytes must be labeled as suspect to alert the data user of the observed problem. Included among those criteria are: response factor check for each PCB calibration congener and retention time reproducibility for SIM data acquisition.

10.3 LABORATORY PERFORMANCE CHECK SOLUTION -- In this method, the medium-level concentration calibration solution also serves the purpose of a laboratory performance check solution.

10.4 LABORATORY SURROGATE SPIKE

10.4.1 Measure the concentration of both surrogate compounds in every sample and blank.

10.4.2 Acceptance limits for surrogate compounds will be 60-140% recovery for sediment extracts.

10.5 LABORATORY CONTROL SAMPLE -- A QC reference check standard (LCS) is also prepared and analyzed for Aroclor-1242 at a concentration of 500 ng/mL in the extract. For sediment/solid samples, sodium sulfate is used for the QC reference check standard (LCS). Calculate the percent recovery for the Total PCB Aroclor spike and compare to the project limits of 60-140%. If the percent recovery for the QC reference check standard (LCS) is out of criteria, the analysis is out of the control for that analyte and the problem should be immediately corrected. The entire batch of samples will need to be reanalyzed (Exception: If the LCS recovery is high and there were no associated positive results, then address the issue in the Case Narrative and no further action is needed).

10.6 MS/MSD SAMPLES -- At this time, the GE Hudson River Design Support Sediment Sampling and Analysis Program does not require the preparation and analysis of matrix spike and/or matrix spike duplicate samples. If requested in the future, a matrix spike for Aroclor-1242 is to be analyzed at a rate of 1 matrix spike per every 20 samples at a concentration of 20,000 ng/mL in the extract (**Note:** this spike concentration will require a sample dilution to be performed). The result by GEHR680 will be reported as a Total PCB. Also a matrix spike duplicate sample is to be analyzed at a rate of 1 per every 20 samples.

10.6.1 If requested, analyze one unspiked and two spiked samples. Calculate the percent recovery based on Aroclor concentration of both samples as follows:

A = concentration of spiked sample
B = concentration of unspiked sample (background)
T = known true value of the spike

Percent Recovery (p) = 100 (A-B) %/T

Compare the percent recovery calculated with the project limits of 60-140%. If the total PCB concentrations of the matrix spikes are *greater* than four times the calculated sample amount, then the quality control limits should be applied. If the total PCB concentrations of the matrix spikes are *less* than four times the sample, then there are no established limits applicable. If the percent recovery falls outside of the acceptance range for the total PCB used as the spiking analyte, then the matrix spike recovery failed the acceptance criteria. Check for documentable errors (*e.g.,* calculations and spike preparations) and then check the unspiked sample results and surrogate recoveries for indications of matrix effects. If no errors are found and the associated QC reference check standard

(Laboratory Control Sample [LCS]) is within 60-140%, then sample matrix effects are the most likely cause. Note this in the Case Narrative.

A relative percent difference (RPD) must also be calculated on the matrix spike set recoveries. This is calculated as follows:

A = % recovery of matrix spike sample
B = % recovery of matrix spike duplicate sample

RPD = [(A-B)/{(A+B)/2}] x 100
where (A-B) *is taken as an absolute value*

If the total PCB concentrations of the matrix spike set are *greater* than four times the calculated sample amount, then an RPD of 40% or less is acceptable. If the total PCB concentrations of the matrix spike set are *less* than four times the calculated sample amount than there are no established limits applicable to the RPD. If the criterion is not met, check for documentable errors (*e.g.,* calculations and spike preparations) and then check the unspiked sample results and surrogate recoveries for indications of matrix effects. If no errors are found and the associated LCS is within 60-140%, then sample matrix effects are the most likely cause. Note this in the Case Narrative.

11. PROCEDURES

11.1 Sediment Samples – The extracts for this analysis will be the same extracts as those generated for the analysis of total PCBs as Aroclors by SOP GEHR8082.

11.2 GC/MS ANALYSIS

11.2.1 Remove the sample extract or blank from storage and allow it to warm to ambient laboratory temperature, if necessary. Add an appropriate volume of the appropriate internal standard stock solution.

11.2.1.1 Internal standard concentration for SIM data acquisition = 4 μL of 40 ng/μL solution (of each chrysene-d_{12} and phenenthrene-d_{10}) added to 200 μL of extract for a concentration of 0.80 ng/μL.

11.2.2 Inject a 2-μL aliquot of the blank/sample extract into the GC operated under conditions used to produce acceptable results during calibration.

11.2.3 Acquire mass spectral data with SIM conditions. Use the same data acquisition time and MS operating conditions previously used to determine response factors.

11.2.4 Examine data for saturated ions in mass spectra of target compounds, if saturation occurred, dilute and reanalyze the extract after the quantity of the internal standards is adjusted appropriately. In addition, any individual PCB analyte amount that exceeds the high level calibration standard of the calibration curve will require dilution and re-analysis of the extract to place that analyte within the calibration range.

11.2.5 For each internal standard, determine that the area measured in the sample extract has not decreased by >30% from the area measured during the most recent previous analysis of a calibration solution or by >50% from the mean area measured during initial calibration. If either criterion is not met, remedial action must be taken to improve sensitivity, and the sample extract must be reanalyzed.

11.3 IDENTIFICATION PROCEDURES

11.3.1 Using the ions shown on Tables 6a-6b for PCBs examine ion current profiles (ICPs) to locate internal standards, surrogate compounds, and PCBs for each isomer group. Use the RRT window data on Table 7 as guidelines for location of PCB isomers. (A reverse search software routine can be used to locate compounds of concern.)

11.3.2 SIM Data -- Obtain appropriate selected ion current profiles (SICPs) for IS quantitation and confirmation ions for the quantitation and confirmation ions for each PCB isomer group.

11.3.3 PCB Analytes

11.3.3.1 For all PCB candidates, confirm the presence of an (M-70)$^-$ ion cluster by examining ICPs or spectra for at least one of the most intense ions in the appropriate ion cluster.

11.3.3.2 For Cl_3-Cl_7 isomer groups, examine ICPs or spectra for intense $(M+70)^+$ ions that would indicate a coeluting PCB containing two additional chlorines. (GC retention time data show that this is not a potential problem for other PCB isomer groups.) If this interference occurs, a correction can be made. Obtain and record the area for the appropriate ion (Table 9) for the candidate PCB isomer group. Use the information in Table 10 to correct the measured abundance of M+. For example, if a Cl_7-PCB and a Cl_5-PCB candidate coelute, the Cl_7-PCB will contribute to the ion measured for m/z 326 and m/z 324, the quantitation and confirmation ions, respectively, for a Cl_5-PCB. Obtain and record the area for m/z 322 (the lowest mass ion in the $(M+-70)^+$ ion cluster of a Cl_5-PCB

fragment produced by a Cl_7-PCB). To determine the m/z 326 and m/z 324 areas produced by the Cl_5-PCB, calculate the Cl_7-PCB contribution to each and subtract it from the measured area. In this example, 164% of the area measured for m/z 322 should be subtracted from the area measured for m/z 324, and 108% of the m/z 322 area should be subtracted from the area measured for m/z 326 (Table 10).

11.3.3.3 For Cl_2-Cl_8-PCB candidates, examine ICPs or spectra for intense $(M+35)^+$ ions that would indicate a coeluting PCB containing one additional chlorine. This coelution causes interferences because of the natural abundance of ^{13}C. (This interference will be small and can be neglected except when measuring the area of a small amount of a PCB coeluting with a large amount of another PCB containing one more chlorine.) To correct for this interference, obtain and record the area for the appropriate ion (Table 11) from the $(M-1)^+$ ion cluster, and subtract 13.5% of the area measured for the $(M-1)^+$ ion from the measured area of the quantitation ion. For example, for Cl_5–PCB candidates, obtain and record the area for m/z 325; subtract 13.5% of that area from the measured area of m/z 326.

11.3.4 All Analytes -- Use ICP data to calculate the ratio of the measured peak areas of the quantitation ion and confirmation ion(s), and compare to the acceptable ratio (Table 9). If acceptable ratios are not obtained, a coeluting or partially coeluting compound may be interfering. Examination of data from several scans may provide information that will allow application of additional background corrections to improve the ion ratio.

11.4 IDENTIFICATION CRITERIA

11.4.1 Internal Standards

11.4.1.1 Chrysene-d_{12} -- the abundance of m/z 241 relative to m/z 240 must be $\geq$ 15% and $\leq$ 25%, and these ions must maximize simultaneously. The area measured for m/z 240 must be within 30% of the area measured during the most recent calibration.

11.4.1.2 Phenanthrene-d_{10} -- the abundance of m/z 189 relative to m/z 188 must be $\geq$ 10% and $\leq$ 22%, and these ions must maximize simultaneously. The area measured for m/z 188 must be within 30% of the area measured during the most recent acceptable calibration.

11.4.1.3 Retention time must be within ±10 s of that observed during the most recent acceptable calibration.

11.4.2 SIM Data for PCBs

11.4.2.1 Absolute retention times of surrogate compounds must be within ±10 s of that measured during the last previous continuing calibration check.

11.4.2.2 Quantitation and confirmation ions for each PCB isomer group must maximize within ±1 scan of each other.

11.4.2.3 The integrated ion current for each quantitation and confirmation ion must be at least three times background noise and must not have saturated the detector.

11.4.2.4 For each PCB isomer group candidate, the ratio of the quantitation ion area to the confirmation ion area must be within limits shown on Table 9; at least one ion in the $(M-70)^+$ ion cluster most be present.

12. CALCULATIONS

12.1 From appropriate ICPs of quantitation ions, obtain and record the spectrum number of the chromatographic peak apex and the area of the entire chromatographic peak.

12.2 GC/MS Analytes Detected Above the Initial Low Level Calibration Standard Concentration

Any individual PCB analyte amount in a sample or QC sample above the initial low-level calibration standard concentration will be reported as such with no associated flags. Any individual PCB analyte amount that exceeds the high level calibration standard of the calibration curve will require dilution and re-analysis of the extract to place that analyte within the calibration range.

12.3 GC/MS Analytes Detected Below the Initial Low Level Calibration Standard Concentration

As stated in Section 1.2, a Method Detection Limit (MDL) will be performed in accordance with procedures set forth in 40CFR Part 136, Appendix B. Any individual PCB analyte amount in a sample or QC sample that is above the established MDL but below the initial low level calibration standard concentration will be reported and appropriately flagged with a "J" flag. A "J" flag signifies that the analyte amount was below the initial low level calibration standard concentration but above the determined MDL for the analyte.

12.4 GC/MS Analytes Detected Below the MDL

Any individual PCB analyte amount in a sample or QC sample that is below the established MDL for that analyte or not present will be reported as not detected (ND). The associated MDL concentration value for that analyte will be reported to provide information on the analyte reporting limit.

12.5 PCB Homolog Group Amounts

For each PCB Homolog Group all reportable (both non-flagged and "J" flagged) PCB analytes associated with a given chlorination level (i.e. All dichlorobiphenyls) will be summed and a total provided. No flagging of Homolog Group concentrations will occur. This will provide 10 PCB sub-totals from monochlorobiphenyl to decachlorobiphenyl. If for a given Homolog there are no reportable analytes to report or sum, then a not detected (ND) will be reported. The associated analyte MDL concentration value for that chlorination level will be reported as the Homolog reporting limit.

12.6 Total PCB Amount

The Total PCB amount for a sample or QC sample will be provided by summation of the Homolog Group amounts. No flagging of the Total PCB amount will occur. If all 10 Homolog Groups are reported as not detected (ND), then the Total PCB amount will be reported as not detected (ND). For this reporting condition (i.e. Total PCB amount = ND), the single highest reporting limit from the 10 Homolog groups (highest PCB analyte MDL from MDL study) will be used and will provide the reporting limit for the Total PCB amount.

12.7 All sediment results will be reported on a dry-weight basis using the moisture determined during the GEHR8082 analysis for total PCBs as Aroclors.

12.8 For PCBs, sum the areas for all isomers identified at each level of chlorination (*e.g.*, sum all quantitation ion areas for Cl_4-PCBs).

12.9 Calculate the concentration of each surrogate compound and PCB isomer group using the formula:

$$C_x = (A_x \cdot Q_{is})/(A_{is} \cdot RF \cdot W \cdot D)$$

where:

C_x = concentration (micrograms per kilogram or micrograms per liter) of surrogate compound or a PCB isomer group,

A_x = the area of the quantitation ion for each surrogate compound or the sum of quantitation ion areas for all PCB isomers at a particular level of chlorination,

A_{is} = the area of the internal standard quantitation ion, m/z 240 for chrysene-d_{12} or m/z 188 for phenanthrene-d_{10},

Q_{is} = quantity (micrograms) of internal standard added to the extract before GC/MS analysis,

RF = calculated response factor for the surrogate compound or the PCB calibration compound for the isomer group (level of chlorination), and

W = weight (kilograms) of sample extracted.

D = (100 - % moisture)/100

12.4.1 Use the average RF calculated during Initial Calibration.

12.4.2 For PCBs, use the RF relative to chrysene-d_{12} unless an interference makes the use of the RF relative to phenanthrene-d_{10} necessary.

12.10 Report calculated values to two significant figures.

12.11 When samples of known composition or fortified samples are analyzed, calculate the percent method bias using the equation:

$$B = 100 (C_s - C_t)/ C_t$$

where:

C_s = measured concentration (in micrograms per kilogram or micrograms per liter) and

C_t = theoretical concentration (*i.e.*, the quantity added to the sample aliquot/weight or volume of sample aliquot).

Note: The bias value retains a positive or negative sign.

13. AUTOMATED IDENTIFICATION AND MEASUREMENT

Automated identification and measurement software for PCBs will be used to assist in handling and reducing the data.

14. REFERENCES

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Table 1. Recommended GC and MS Operating Parameters

Mass Spectrometer Parameters:

Varian Model Saturn 2000 Ion Trap Mass Spectrometer

Ion Trap Temperature: 200°C
Emission Current: 70 microamps
Scan Time: 0.700 seconds
Ionization Mode: EI AGC
Ion Technique: SIS (Selected Ion scanning, which is Varian's term for SIM)

Gas Chromatograph Parameters:

Varian Model 3800 Gas Chromatograph

Injector Model 1079 (Temperature Programmable):

Coolant: Enabled
Initial Temperature: 50°C
Initial Hold Time: 1.00 minute
Final Temperature: 250°C
Temperature Ramp: 150°C/minute
Final Hold Time: 50 minutes
1079 Valve at 0.0 minutes: split state: on split ratio: 50
1079 valve at 0.50 minutes: split state: off split ratio: off
1079 valve at 3.00 minutes: split state: on split ratio: 50
Constant column flow: 1.0mL/min
Pulsed Pressure: On
Pulse Pressure: 24.0 psi
Pulse Pressure Duration: 0.75 minutes

Oven Temperature Profile:

Initial Temperature: 80°C
Initial Hold Time: 1.00 minute
First Temperature Ramp: 80 °C to 160°C at 30°C/minute, hold 1 minute
Second Temperature Ramp: 160°C to 310°C at 3°C/minute, hold 5.30 minutes

Table 1. Recommended GC and MS Operating Parameters (Cont.)

Sample Injection Parameters:

Syringe Size: 10uL
Solvent Flush: Yes, solvent A then B
Syringe Wash: 20 seconds
Solvent plug size: 0.3uL
Upper Air Gap: Yes
Lower Air Gap: Yes
Injection Rate: 0.5uL/sec
Extract Injection Volume: 2.0uL

Table 2. PCB Congeners Used as Calibration Standards

PCB Isomer Group	Congener Number [a]	Chlorine Substitution
Concentration Calibration Standard		
Monochlorobiphenyl	1	2
Dichlorobiphenyl	5	2, 3
Trichlorobiphenyl	29	2, 4, 5
Tetrachlorobiphenyl	50	2, 2', 4, 6
Pentachlorobiphenyl	87	2, 2', 3, 4, 5'
Hexachlorobiphenyl	154	2, 2', 4, 4', 5, 6'
Heptachlorobiphenyl	188	2, 2', 3, 4', 5, 6, 6'
Octachlorobiphenyl	200	2, 2', 3, 3', 4, 5', 6, 6'
Nonachlorobiphenyl[b]	-	---
Decachlorobiphenyl	209	2, 2', 3, 3', 4, 4', 5, 5', 6, 6'
Retention Time Calibration Standards		
Tetrachlorobiphenyl	77	3, 3', 4, 4'
Pentachlorobiphenyl	104	2, 2', 4, 6, 6'
Heptachlorobiphenyl	189	2, 3, 3′ 4, 4′, 5, 5′
Octachlorobiphenyl	202	2, 2′, 3, 3′, 5, 5′, 6, 6′

[a] Numbered according to the system of Ballschmiter and Zell (2).
[b] Decachlorobiphenyl is used as the calibration congener for both nona- and decachlorobiphenyl isomer groups.

Table 3. Scheme for Preparation of PCB Primary Dilution Standards

PCB Cong.	Isomer Group	Stock Sol. Conc. mg/mL	Proportion for Primary Dil. Sol.	Primary Dil. Std. Conc. ng/μL
#1	Cl_1	1.0	1 part	50
#5	Cl_2	1.0	1 part	50
#29	Cl_3	1.0	1 part	50
#50	Cl_4	1.0	2 parts	100
#87	Cl_5	1.0	2 parts	100
#154	Cl_6	1.0	2 parts	100
#188	Cl_7	1.0	3 parts	150
#200	Cl_8	1.0	3 parts	150
#209	Cl_{10}	1.0	5 parts	250
			Total 20 parts	

Table 4a. Composition and Approximate Concentrations of Calibration Solutions for SIM Data Acquisition for PCB Determinations

Concentration (ng/μL)

Compound	CAL 1	CAL 2	CAL 3	CAL 4	CAL 5	CAL 6
Cal. Congeners						-
Cl_1 (#1)	0.05	0.5	1	2	5	-
Cl_2 (#5)	0.05	0.5	1	2	5	-
Cl_3 (#29)	0.05	0.5	1	2	5	-
Cl_4 (#50)	0.10	1.0	2	4	10	-
Cl_5 (#87)	0.10	1	2	4	10	-
Cl_6 (#154)	0.10	1	2	4	10	-
Cl_7 (#188)	0.15	1.5	3	6	15	-
Cl_8 (#200)	0.15	1.5	3	6	15	-
Cl_{10} (#209)	0.25	2.5	5	10	-	15
Internal Standards						
Chrysene-d_{12}	0.80	0.80	0.80	0.80	0.80	0.80
Phenanthrene-d_{10}	0.80	0.80	0.80	0.80	0.80	0.80
Surrogate Compounds						
TCMX	0.05	0.5	1	2	5	-
DCB	0.05	0.5	1	2	5	-

Table 4b. Retention Time Calibration Standards
Concentration (ng/μL)

Compound	CAL 1
Pentachlorobiphenyl (#014)	2.50
Tetrachlorobiphenyl (#77)	2.50
Octachlorobiphenyl (#202)	2.50
Heptachlorobiphenyl (#189)	2.50

Table 5. Criteria for DFTPP Spectrum

m/z	Relative Abundance
127	40-60%
197	<1%
198	100% (Base Peak)
199	5-9%
275	10-30%
365	>1%
441	Present and <m/z 443
442	>40%
443	17-23% of m/z 442

Table 6a. Ions for Selected Ion Monitoring to Determine PCBs by Acquiring Data for Five Sets of ≤27 Ions Each

Ion Set	Isomer Group/ IS/Surrogate	Quant. Ion	Confirm Ions	M-70 Ions	M+70 Ions	M+35 Ions	Ion Measured[a] for Correction	
1	Cl_1	188	190	152, 153[b]	256, 258	222, 224	-	-
	Cl_2	222	224	152, 153, 186, 188[c]	290, 292, 294	256, 258	-	221
	Cl_3	256	258	186, 188	-	290, 292, 294	-	225
	Cl_4	292	290, 294	220, 222	-	-	-	-
	Phenanthrene-d_{10}	188	189	-	-	-	-	-
2	Cl_3	256	258	188, 188	324, 326, 328	290, 292, 294	254	255
	Cl_4	292	290, 294	220, 222	360, 362	324, 326, 328	288	289
	Cl_5	326	324, 328	254, 256, 258	-	360, 362	-	323
	Cl_6	360	358, 362	288, 290, 292	-	-	-	-
3	Cl_5	326	324, 328	254, 256	392, 394, 396, 398	360, 362	322	323
	Cl_6	360	358, 362	288, 290	-	392, 394, 396, 398	-	357
	Cl_7	394	392, 396	322, 324, 326	-	-	-	-
4	Cl_6	360	358, 362	288, 290	426, 428, 430, 432	392, 394, 396	356	357
	Cl_7	394	392, 396, 398	322, 324	-	428, 430, 432	-	391
	Cl_8	430	428, 432	356, 358, 360	-	462, 464, 466	-	425
	Cl_9	464	460, 462, 466	390, 392, 394	-	-	-	-
	Chrysene-d_{12}	240	241	-	-	-	-	-

Table 6a. Ions for Selected Ion Monitoring to Determine PCBs by Acquiring Data for Five Sets of ≤27 Ions Each (Cont.)

Ion Set	Isomer Group/ IS/Surrogate	Quant. Ion	Confirm Ions	M-70 Ions	M+70 Ions	M+35 Ions	Ion Measured[a] for Correction	
5	Cl_8	430	426, 428, 432	356, 358, 360	494, 496, 498, 500	462, 464, 466	-	425
	Cl_9	464	460, 462, 466	390, 392, 394	-	496, 498, 500	-	-
	Cl_{10}	498	494, 496, 500	424, 426, 428, 430	-	-	-	-

[a]See Tables 9-10.
[b]Cl_1-PCBs lose HCl.
[c]Some Cl_2-PCBs lose Cl_2 and some lose HCl.

Table 6b. Ions for Selected Ion Monitoring to Determine PCBs by Acquiring Data for Five Ion Sets of ≤27 Ions

Ion Set #1[a]	Ion Set #2[b]	Ion Set #3[c]	Ion Set #4[d]	Ion Set #5[e]
152	186	247	240	356
153	188	249	241	358
186	220	254	288	360
187	222	256	290	390
188	254	288	322	392
189	255	290	324	394
190	256	322	326	424
220	258	323	356	425
221	288	324	357	426
222	289	326	358	428
224	290	328	360	430
255	292	357	362	432
256	294	358	390	462
258	323	360	391	464
290	324	362	392	466
292	326	392	394	496
294	328	394	396	498
	358	396	398	499
	360	398	425	500
	362		426	502
			428	
			430	
			432	
			460	
			462	
			464	
			466	

Table 6b. Ions for Selected Ion Monitoring to Determine PCBs by Acquiring Data for Five Ion Sets of ≤27 Ions (Cont.)

Scan Range #1	Scan Range #2	Scan Range #3	Scan Range #4	Scan Range #5
145m/z to 330m/z	179m/z to 398m/z	240m/z to 428m/z	233m/z to 520m/z	349m/z to 532m/z
SIS Ion Preparation	**SIS Ion Preparation**	**SIS Ion Preparation**	**SIS Ion Preparation**	**SIS Ion Preparation**
Mass Range 1: 150mz/ to 155m/z	Mass Range 1: 184mz/ to 226m/z	Mass Range 1: 244mz/ to 260m/z	Mass Range 1: 236mz/ to 296m/z	Mass Range 1: 350mz/ to 366m/z
Mass Range 2: 180mz/ to 195m/z	Mass Range 2: 250mz/ to 263m/z	Mass Range 2: 284mz/ to 294m/z	Mass Range 2: 316mz/ to 368m/z	Mass Range 2: 384mz/ to 400m/z
Mass Range 3: 215mz/ to 260m/z	Mass Range 3: 283mz/ to 300m/z	Mass Range 3: 318mz/ to 332m/z	Mass Range 3: 385mz/ to 404m/z	Mass Range 3: 418mz/ to 438m/z
Mass Range 4: 285mz/ to 300m/z	Mass Range 4: 320mz/ to 332m/z	Mass Range 4: 388mz/ to 404m/z	Mass Range 4: 422mz/ to 438m/z	Mass Range 4: 456mz/ to 472m/z
Mass Range 5: not used	Mass Range 5: 355m/z to 368m/z	Mass Range 5: not used	Mass Range 5: 455m/z to 506m/z	Mass Range 5: 490m/z to 508m/z
R.T. Window (minutes)	**R.T. Window (minutes)**	**R.T. Window (minutes)**	**R.T. Window (minutes)**	**R.T. Window (minutes)**
5.00 to 17.21	17.21 to 25.01	25.01 to 30.25	30.25 to 36.35	36.35 to 54.67
Total number of Ions	**Total number of Ions**	**Total number of Ions**	**Total number of Ions**	**Total number of Ions**
17	20	19	27	20

[a] Ions to identify and measure Cl_1 – Cl_4 PCBs and phenanthrene-d_{10}.

[b] Ions to identify and measure Cl_3– Cl_6 PCBs.

[c] Ions to identify and measure Cl_5 – Cl_7 PCBs.

[d] Ions to identify and measure Cl_6 – Cl_9 PCBs and chrysene-d_{12}.

[e] Ions to identify and measure Cl_8 – Cl_{10} PCBs.

Table 7. Retention Time Data for PCB Isomer Groups and Calibration Congeners

Isomer Group	Approximate RRT Range[a]	Cal. Cong. Number	Cal. Cong. RRT[a]
Monochlorobiphenyls	0.23-0.28	1	0.23
Dichlorobiphenyls	0.29-0.43	5	0.34
Trichlorobiphenyls	0.36-0.60	29	0.45
Tetrachlorobiphenyls	0.44-0.77	50	0.47
Pentachlorobiphenyls	0.55-0.93	87	0.73
Hexachlorobiphenyls	0.65-1.07	154	0.74
Heptachlorobiphenyls	0.81-1.13	188	0.81
Octachlorobiphenyls	0.95-1.18	200	0.97
Nonachlorobiphenyls	1.11-1.22	-	-
Decachlorobiphenyls	1.26	209	1.26

[a] Retention time relative to chrysene-d_{12} with a 30 m $\times$ 0.25 mm ID DB-XLB fused silica capillary column and the GC conditions set forth in Table 1.

Table 8. Known Relative Abundances of Ions in PCB Molecular Ion Clusters[a]

m/z	Relative Intensity
Monochlorobiphenyls	
188	100.00
189	13.50
190	33.40
192	4.41
Dichlorobiphenyls	
222	100.00
223	13.50
224	66.00
225	8.82
226	11.20
227	1.44
Trichlorobiphenyls	
256	100.00
257	13.50
258	98.60
259	13.20
260	32.70
261	4.31
262	3.73
263	0.47
Tetrachlorobiphenyls	
290	76.20
291	10.30
292	100.00
293	13.40
294	49.40
295	6.57
296	11.00
297	1.43
298	0.95
Pentachlorobiphenyls	
324	61.00
325	8.26
326	100.00
327	13.50
328	65.70
329	8.78
330	21.70
331	2.86
332	3.62
333	0.47
334	0.25
Hexachlorobiphenyls	
358	50.90
359	6.89
360	100.00
361	13.50
362	82.00
363	11.00
364	36.00
365	4.77
366	8.92
367	1.17
368	1.20
369	0.15
Heptachlorobiphenyls	
392	43.70
393	5.91
394	100.00
395	13.50
396	98.30
397	13.20
398	53.80
399	7.16
400	17.70
401	2.34
402	3.52
403	0.46
404	0.40
Octachlorobiphenyl	
426	33.40
427	4.51
428	87.30
429	11.80
430	100.00
431	13.40
432	65.6
433	8.76
434	26.90
435	3.57
436	7.10
437	0.93
438	1.18
439	0.15
440	0.11
Nonachlorobiphenyls	
460	26.00
461	3.51
462	76.40
463	10.30
464	100.00
465	13.40
466	76.40
467	10.20
468	37.60
469	5.00
470	12.40
471	1.63
472	2.72
473	0.35
474	0.39
Decachlorobiphenyl	
494	20.80
495	2.81
496	68.00
497	9.17
498	100.00
499	13.4
500	87.30
501	11.70
502	50.00
503	6.67
504	19.70
505	2.61
506	5.40
507	0.71
508	1.02
509	0.13

[a] Source: Rote and Morris (7)

Table 9. Quantitation, Confirmation, and Interference Check Ions for PCBs, Internal Standards, and Surrogate Compounds

Analyte/ Internal Std.	Nom. MW	Quant. Ion	Confirm. Ion	Expected Ratio[a]	Accept Ratio[a]	M-70 Confirm. Ion	Interference Check Ions M+70	Interference Check Ions M+35
PCB Isomer Group								
Cl_1	188	188	190	3.0	2.5-3.5	152[b]	256	222
Cl_2	222	222	224	1.5	1.3-1.7	152	292	256
Cl_3	256	256	258	1.0	0.8-1.2	186	326	290
Cl_4	290	292	290	1.3	1.1-1.5	220	360	326
Cl_5	324	326	324	1.6	1.4-1.8	254	394	360
Cl_6	358	360	362	1.2	1.0-1.4	288	430	394
Cl_7	392	394	396	1.0	0.9-1.2	322	464	430
Cl_8	426	430	428	1.1	0.9-1.3	356	498	464
Cl_9	460	464	466	1.3	1.1-1.5	390	-	498
Cl_{10}	494	498	500	1.1	0.9-1.3	424	-	-
Internal standards								
Chrysene-d_{12}	240	240	241	5.1	4.3-5.9	-	-	-
Phenanthrene-d_{10}	188	188	189	6.6	6.0-7.2	-	-	-
Surrogate compounds								
TCMX	242	244	242	1.3	1.1-1.5	-	-	-
DCB	494	498	500	1.1	0.9-1.3	424	-	-

[a] Ratio of quantitation ion to confirmation ion.

[b] Monochlorobiphenyls lose HCl to produce an ion at m/z 152.

Table 10. Correction for Interference of PCB Containing Two Additional Chlorines

Candidate Isomer Group	Quant. Ion	Confirm. Ion	Ion Measured to Determine Interference	% of Meas. Ion Area to be Subtracted from	
				Quant. Ion Area	Confirm. Ion Area
Trichlorobiphenyls	256	258	254	99%	33%
Tetrachlorobiphenyls	292	290	288	65%	131%
Pentachlorobiphenyls	326	324	322	108%	164%
Hexachlorobiphenyls	360	362	356	161%	71%
Heptachlorobiphenyls	394	396	390	225%	123%

Table 11. Correction for Interference of PCB Containing One Additional Chlorine

Candidate Isomer Group	Quant. Ion	Ion Measured to Determine Interference	% of Meas. Ion Area to be Subtracted from Quant. Ion Area
Dichlorobiphenyls	222	221	13.5%
Trichlorobiphenyls	256	255	13.5%
Tetrachlorobiphenyls	292	289	17.4%
Pentachlorobiphenyls	326	323	22.0%
Hexachlorobiphenyls	360	357	26.5%
Heptachlorobiphenyls	394	391	30.9%
Octachlorobiphenyls	430	425	40.0%

Figure 1. Merged Ion Current Profile of PCB Calibration Congeners

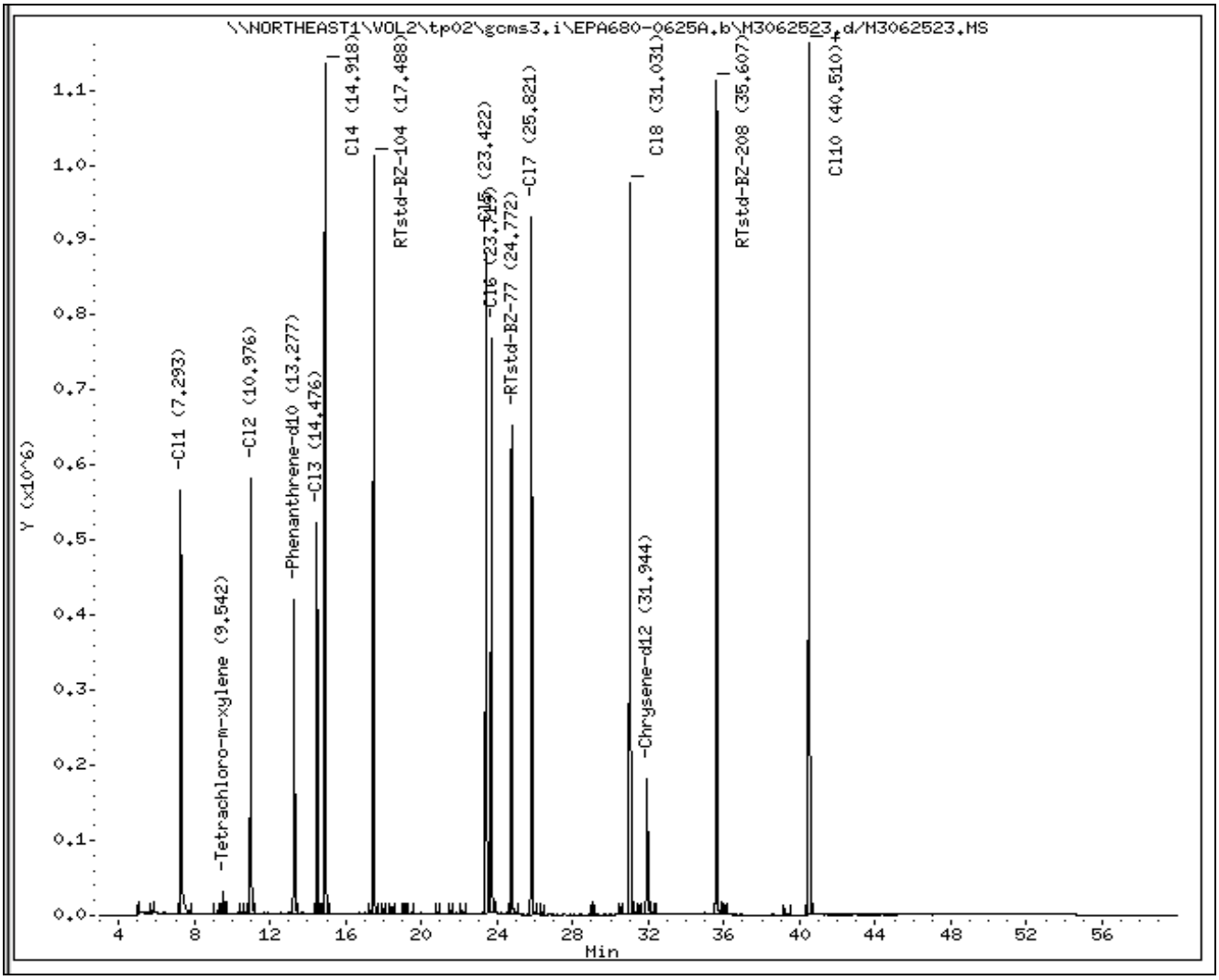
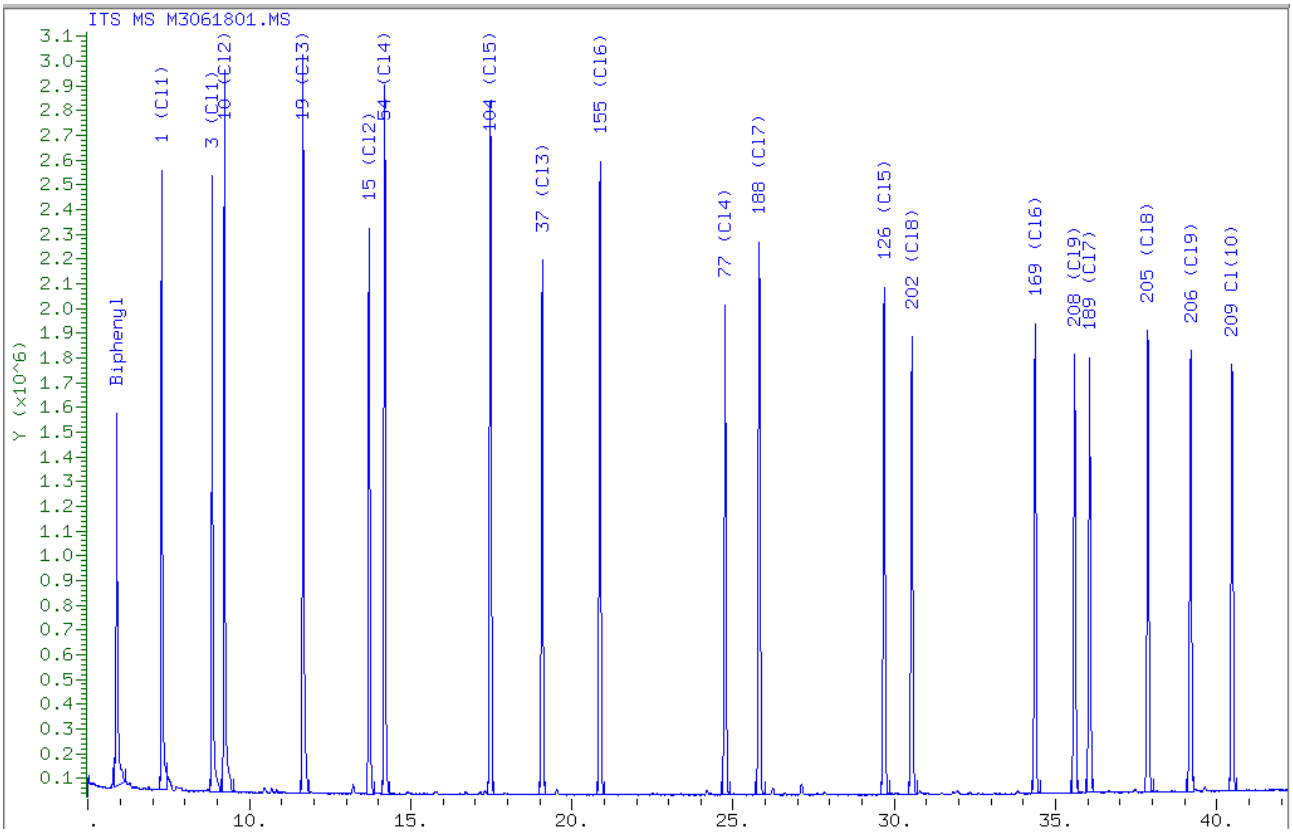


Figure 2: Total Ion Chromatogram of PCB Window Defining Mixture Standard



APPENDIX 9

OBJECTIVE

This Standard Operating Procedure (SOP) describes the requirements for the data packages that will be generated as part of the Hudson River Design Support Sediment Sampling and Analysis Program. This SOP applies to the contractor(s) involved in analytical data generation and reporting. All data packages generated for the Hudson River Design Support Sediment Sampling and Analysis Program must be provided in an Adobe Acrobat (.PDF) file format. The laboratory will be notified of the samples that will undergo data validation. For these samples, the laboratory will be required to generate hard copy data packages as well as the Adobe Acrobat (.PDF) file format.

SECTION A-9 DATA PACKAGE DELIVERABLES

The following sections describe in detail the types of data packages designed for the Hudson River Design Support Sediment Sampling and Analysis Program. These details are provided to allow several participating laboratories to produce data packages that are similar in format, order of presentation, and content. The data packages detailed in Section A-9.1 have been developed based on deliverables specified in the US EPA Contract Laboratory Program Statement of Work (CLP SOW). The CLP SOW has additional details concerning data packages that are specific to the CLP analyses. The most recent Statement of Work should be referenced for details concerning CLP-style data packages. Note: the summary forms provided in these data packages should be in similar format and content to the Contract Laboratory Program (CLP) forms listed (as references) next to the form title. These CLP forms references are only provided as guidance on content and format and should be modified by the laboratory to meet specific method requirements. Section A-9.2 provides details concerning specific contents of the data deliverables described in Section A-9.1.

The data package deliverables are categorized into two distinct levels as follows:

Level A - Case Narrative, analytical results, and Chain-of-Custody Records for the sample delivery group (SDG).

Level B - Fully documented data package.

The Level A data package is a basic "results-only" style of data package that includes a cover letter, SDG narrative, field Chain-of-Custody Records, analytical results summaries, and a glossary of qualifier codes. The Level B package resembles the information required by the CLP SOW. This type of package includes all information provided in Level A package but also includes summary forms for quality control procedures and all sample and quality control raw data to support the results reported.

A-9.1 Data Package Contents and Order of Presentation

The laboratory will be required to submit supporting documentation for the reported analytical results. The supporting documentation and the analytical results will be reported in one of two data package delivery categories. The categories are defined below. The data package deliverables must be submitted in the order in which the deliverables appear in the text. The laboratory need not include the documentation for any fraction not required for an SDG.

A-9.1.1 General Format for Level B Deliverables

For some analyses, Level B Sample Data Package deliverables may be requested. A Level A Data Package will also be required with the Level B package as a summary package.

The Level B Sample Data Package shall include data for analyses of all samples in one SDG, including field samples, reanalyses, secondary dilutions, blanks, laboratory control samples, matrix spikes, matrix spike duplicates, and/or laboratory duplicates. The complete Sample Data Package is divided into the units as described below. Units for each analytical fraction have been detailed. If the analysis of that fraction was not required for samples in the SDG, the fraction-specific unit is not a required deliverable. The Sample Data Package must be complete before submission and must be consecutively paginated. The Sample Data Package will be arranged in the following order:

A) Cover Letter/Letter of Transmittal signed by the laboratory manager.

B) Title Page

C) Table of Contents

D) Sample Delivery Group (SDG) Narrative

This document shall be clearly labeled "SDG Narrative" and shall contain: laboratory name; SDG number; GE sample identifications; laboratory sample numbers; and detailed documentation of any quality control, sample, shipment, and/or analytical problems encountered in processing (preparing and analyzing) the samples reported in the data package. A glossary of qualifier codes used in the SDG must also be provided.

The laboratory must also include any technical and administrative problems encountered, corrective actions taken and method of resolution, and an explanation of all flagged edits (i.e., exhibit edits) on quantitation reports.

Additionally, the SDG Narrative must be signed and dated by the laboratory manager.

E) Field and Internal (Laboratory) Chain-of-Custody Records and Sample Receipt Documentation Log

Copies of both the external and internal Chain-of-Custody Records for all samples within the SDG must be included in the deliverables. A description of the condition and temperature of the samples upon laboratory receipt (*i.e.*, custody seal condition, container status) must be provided for each Chain-of-Custody Record/sample cooler.

F) GC/MS Volatile Organic Data.

1. Quality Control (QC) Summary.

a. Surrogate Percent Recovery Summary (modified CLP SOW288 Form II VOA).

b. Matrix Spike/Matrix Spike Duplicate Summary (modified CLP SOW288 Form III VOA).

c. Laboratory Control Sample Summary (modified CLP SOW288 Form III VOA).

d. Method Blank Summary (modified CLP SOW288 Form IV VOA) -- arranged in chronological order by date of analysis of the blank, by instrument.

e. GC/MS Tuning and Mass Calibration Summary (modified CLP SOW288 Form V VOA) -- arranged in chronological order, by instrument.

f. Internal Standard Area and Retention Time Summary (modified CLP SOW288 Form VIII VOA) -- arranged in chronological order, by instrument.

2. Sample Data

Sample data shall be arranged in packets consisting of the Analytical Results Summaries followed by the raw data for volatile samples. These sample packets should then be placed in increasing alphanumeric order by GE sample identification. The order of each sample packet is as follows:

a. Target Compound Results (modified CLP SOW288 Form I VOA).

b. Reconstructed total ion chromatogram (RIC) and quantitation reports.

c. Copies of raw spectra and copies of background-subtracted mass spectra of each target compound identified in the sample and corresponding background-subtracted target compound standard mass spectra.

d. Exhibit work sheet (including example calculations showing how sample results are calculated using the initial calibration and sample responses for at least one sample).

3. Standards Data

a. Initial Calibration Data (modified CLP SOW288 Form VI VOA and associated volatile standard RICs and quantitation reports) -- for all initial calibrations associated with analyses in the SDG, in chronological order, by instrument. If a curve equation is utilized, the laboratory must provide the curve equation and coefficient of determination.

b. Continuing Calibration Data (modified CLP SOW288 Form VII VOA and associated volatile standard RICs and quantitation reports) -- for all continuing calibrations associated with analyses in the SDG, in chronological order, by instrument.

4. Raw QC Data

a. For each GC/MS tuning and mass calibration (in chronological order, by instrument):

1. Bromofluorobenzene (BFB) bar graph spectrum.

2. BFB mass listing.

b. Method/Storage Blank Data - in chronological order, by instrument:

i. Target Compound Results (modified CLP SOW288 Form I VOA).

ii. RIC and quantitation reports.

iii. Copies of raw spectra and copies of background-subtracted mass spectra of each target compounds identified in the blank and corresponding background-subtracted target compound standard mass spectra.

c. Laboratory Control Sample Data:

i. Target Compound Results (modified CLP SOW288 Form I VOA).

ii. RIC and quantitation reports.

d. Matrix Spike Data:

i. Target Compound Results (modified CLP SOW288 Form I VOA).

ii. RIC and quantitation reports.

e. Matrix Spike Duplicate Data:

i. Target Compound Results (modified CLP SOW288 Form I VOA).

ii. RIC and quantitation reports.

G) GC/MS Semivolatile Organic Data

1. QC Summary

a. Surrogate Percent Recovery Summary (modified CLP SOW288 Form II SV).

b. Matrix Spike/Matrix Spike Duplicate Summary (modified CLP SOW288 Form III SV).

c. Laboratory Control Sample Summary (modified CLP SOW288 Form III SV).

d. Method Blank Summary (modified CLP SOW288 Form IV SV) -- arranged in chronological order by date of analysis of the blank, by instrument.

e. GC/MS Tuning and Mass Calibration Summary (modified CLP SOW288 Form V SV) -- arranged in chronological order, by instrument.

f. Internal Standard Area and Retention Time Summary (modified CLP SOW288 Form VIII SV-1, SV-2) -- arranged in chronological order, by instrument.

2. Sample Data

Sample data shall be arranged in packets consisting of the Analytical Results Summaries, followed by the raw data for semivolatile samples. These sample packets should then be placed in increasing alphanumeric order by GE sample identification. The order of each sample packet is as follows:

a. Target Compound Results (modified CLP SOW288 Form I SV-1, SV-2).

b. RIC and quantitation report.

c. Copies of raw spectra and copies of background-subtracted mass spectra of each target compound identified in the sample and corresponding background-subtracted target compound standard mass spectra.

d. UV traces from Gel Permeation Chromatography (GPC) chromatograms cleanup (if performed).

e. Exhibit work sheet (including example calculations showing how sample results are calculated using the initial calibration and sample responses for at least one sample).

3. Standards Data

   a. Initial Calibration Data (modified CLP SOW288 Form VI SV-1, SV-2 and associated semivolatile standard RICs and quantitation reports) -- for all initial calibrations associated with analyses in the SDG, in chronological order, by instrument. If a curve equation is utilized, the laboratory must provide the curve equation and coefficient of determination.

   b. Continuing Calibration Data (modified CLP SOW288 Form VII SV-1, SV-2 and associated semivolatile standard RICs and quantitation reports) -- for all continuing calibrations associated with analyses in the SDG, in chronological order, by instrument.

4. Raw QC Data

   a. For each GC/MS tuning and mass calibration (in chronological order, by instrument):

      i. Decafluorotriphenylphosphine (DFTPP) bar graph spectrum.

      ii. DFTPP mass listing.

   b. Blank Data -- in chronological order, by instrument:

      i. Target Compound Results (modified CLP SOW288 Form I SV-1, SV-2).

ii. RIC and quantitation reports.

iii. Copies of raw spectra and copies of background-subtracted mass spectra of each target compounds identified in the blank and corresponding background-subtracted target compound standard mass spectra.

c. Laboratory Control Sample Data:

i. Target Compound Results (modified CLP SOW288 Form I SV-1, SV-2).

ii. RIC and quantitation reports.

d. Matrix Spike Data:

i. Target Compound Results (modified CLP SOW288 Form I SV-1, SV-2).

ii. RIC and quantitation reports.

e. Matrix Spike Duplicate Data

i. Target Compound Results (modified CLP SOW288 Form I SV-1, SV-2).

ii. RIC and quantitation reports.

H) GC Organochlorine Pesticide Data

1. QC Summary

a. Surrogate Percent Recovery Summary (modified CLP SOW288 Form II PEST).

b. Matrix Spike/Matrix Spike Duplicate Summary (modified CLP SOW288 Form III PEST).

c. Laboratory Control Sample Summary (modified CLP SOW288 Form III PEST).

d. Method Blank Summary (modified CLP SOW288 Form IV PEST) -- arranged in chronological order by date of analysis of the blank, by instrument.

2. Sample Data

Sample data shall be arranged in packets consisting of the Analytical Results Summaries followed by the raw data for organochlorine pesticide samples. These sample packets should then be placed in increasing alphanumeric order by GE sample identification. The order of each sample packet is as follows:

a. Analytical Results Summary (modified CLP SOW288 Form I PEST).

b. Copies of organochlorine pesticide chromatograms.

c. Copies of organochlorine pesticide chromatograms from second GC column confirmation (if performed).

d. GC integration reports or data system printouts.

e. Exhibit work sheet (including example calculation showing how sample results are calculated using initial calibration standard and sample responses for at least one sample).

f. UV traces from GPC cleanup (if performed).

g. If organochlorine pesticides are confirmed by GC/MS, the laboratory must submit copies of raw spectra and copies of background-subtracted mass spectra of target compounds that are identified in the sample and corresponding background-subtracted target compound standard mass spectra. For multi-component pesticides confirmed by GC/MS, the laboratory will submit mass spectra of three major peaks of multi-component compounds from samples and standards.

3. Standards Data

a. Analytical Sequence Form -- in chronological order, by GC column, by instrument for all samples and quality control analyses.

b. Initial Calibration Data (Initial Calibration Summary Form [inclusive of retention time windows, calibration factors, %RSDs, %Ds, etc.], organochlorine pesticide standard chromatograms, and integration reports) -- for each initial calibration associated with SDG in chronological order, by GC column, by instrument. If a curve equation is utilized, the laboratory must provide the curve equation and coefficient of determination.

c. Continuing Calibration Data (Continuing Calibration Summary Form [inclusive of retention time windows, calibration factors, %RSDs, %Ds, etc.], organochlorine pesticide standard chromatograms, and integration reports) -- for each continuing calibration associated with SDG in chronological order, by GC column, by instrument following the associated initial calibrations.

d. 4,4′-DDT and Endrin Breakdown Data (Percent Breakdown Summary Form, organochlorine pesticide chromatograms and integration reports) -- for each standard associated with SDG in chronological order by GC column, by instrument.

4. Raw QC Data

a. Blank Data -- in chronological order, by instrument:

i. Target Compound Results (modified CLP SOW288 Form I PEST).

ii. Organochlorine pesticide chromatograms and integration reports.

b. Laboratory Control Sample Data:

i. Target Compound Results (modified CLP SOW288 Form I PEST).

ii. Organochlorine pesticide chromatograms and integration reports.

c. Matrix Spike Data:

i. Target Compound Results (modified CLP SOW288 Form I PEST).

ii. Organochlorine pesticide chromatograms and integration reports.

d. Matrix Spike Duplicate Data:

i. Target Compound Results (modified CLP SOW288 Form I PEST).

ii. Organochlorine pesticide chromatograms and integration reports.

e. UV traces from GPC cleanup (if performed).

i. UV traces for the initial calibration standards and blanks. Compound names shall be written over the peaks or printed over the peaks, or retention times shall be written over the peaks, and a separate table listing compounds and retention times shall be provided.

ii. Chromatographs and data system reports for all standards used to quantify compounds in the GPC blanks.

iii. Chromatographs and data system reports for the GPC calibration check solution and all standards used to quantify compounds in the GPC calibration check solution.

f. Raw Florisil® data, arranged in chronological order.

i. Chromatographs and data system reports for the analysis of the Florisil® cartridge performance check.

ii. Chromatographs and data system reports for the standards used to quantify compounds in the Florisil® cartridge performance check analysis (*i.e.*, INDA, INDB, and the 2,4,5-trichlorophenol standards).

I) GC Polychlorinated Biphenyl (PCB) Data

1. QC Summary

a. Surrogate Percent Recovery Summary (modified CLP SOW288 Form II PEST).

b. Matrix Spike/Matrix Spike Duplicate Summary (modified CLP SOW288 Form III PEST).

c. Laboratory Control Sample Summary (modified CLP SOW288 Form III PEST).

d. Method Blank Summary (modified CLP SOW288 Form IV PEST) -- arranged in chronological order by date of analysis of the blank, by instrument.

2. Sample Data

Sample data shall be arranged in packets consisting of the Analytical Results Summaries followed by the raw data for PCB samples. These sample packets should then be placed in increasing alphanumeric order by GE sample identification. The order of each sample packet is as follows:

a. Analytical Results Summary (modified CLP SOW288 Form I PEST).

b. Copies of PCB chromatograms.

c. Copies of PCB chromatograms from second GC column confirmation (if performed).

d. GC integration reports or data system printouts. The integration reports or data system printouts must include all peaks not just the peaks corresponding to the target analytes.

e. Exhibit work sheets (including example calibration showing how sample results are calculated using initial calibration and sample responses for at least one sample).

f. UV traces from GPC (if performed).

g. If PCBs are confirmed by GC/MS, then the laboratory must submit copies of raw spectra and background-subtracted mass spectra of target compounds that are identified in the sample and corresponding background-subtracted target compound standard mass spectra. The laboratory will submit mass spectra of three major peaks of multi-component compounds from samples and standards for each PCB result confirmed by GC/MS.

3. Standards Data

a. Analytical Sequence Form -- in chronological order, by GC column, by instrument for all samples and quality control analyses.

b. Initial Calibration Data -- Initial Calibration Summary Form (inclusive of retention time windows, calibration factors, %RSDs, %Ds, etc.), PCB standard chromatograms, and integration reports for each initial calibration associated with SDG in chronological order, by GC column, by instrument. If a curve equation is utilized, the laboratory must provide the curve equation and coefficient of determination.

c. Continuing Calibration Data -- Continuing Calibration Summary Form (inclusive of retention time windows, calibration factors, %RSDs, %Ds, etc.), PCB standard chromatograms, and integration reports for each continuing calibration associated with SDG in chronological order, by GC column, by instrument following the associated initial calibration.

4. Raw QC data

a. Blank Data -- in chronological order, by instrument:

i. Target Compound Results (modified CLP SOW288 Form I PEST).

ii. PCB chromatograms and integration reports.

b. Laboratory Control Sample Data:

i. Target Compound Results (modified CLP SOW288 Form I PEST).

ii. PCB chromatograms and integration reports.

c. Matrix Spike Data:

i. Target Compound Results (modified CLP SOW288 Form I PEST).

ii. PCB chromatograms and integration reports.

d. Matrix Spike Duplicate Data:

i. Target Compound Results (modified CLP SOW288 Form I PEST).

ii. PCB chromatograms and integration reports.

e. UV traces from GPC cleanup (if performed).

i. UV traces for the initial calibration standards and blanks. Compound names shall be written or printed over the peaks, or retention times shall be written over the peaks, and a

separate table listing compounds and retention times shall be provided.

ii. Chromatographs and data system reports for all standards used to quantify compounds in the GPC blanks.

iii. Chromatographs and data system reports for the GPC calibration check solution and all standards used to quantify compounds in the GPC calibration check solution (or used to assess the Aroclor pattern).

f. Raw Florisil® data, arranged in chronological order:

i. Chromatographs and data system reports for the analysis of the Florisil® cartridge performance check.

ii. Chromatographs and data system reports for the standards used to quantify compounds in the Florisil® cartridge performance check analysis (*i.e.*, INDA, INDB, and the 2,4,5-trichlorophenol standards).

J) GC Herbicide Data

1. QC Summary

a. Surrogate Percent Recovery Summary ("CLP SOW288-like" Form II PEST).

b. Matrix Spike/Matrix Spike Duplicate Summary ("CLP SOW288-like" Form III PEST).

c. Laboratory Control Sample Summary ("CLP SOW288-like" Form III PEST).

d. Method Blank Summary ("CLP SOW288-like" Form IV PEST) -- arranged in chronological order by date of analysis of the blank, by instrument.

2. Sample Data

Sample data shall be arranged in packets consisting of the Analytical Results Summaries followed by the raw data for herbicide samples. These sample packets should then be placed in increasing alphanumeric order by GE sample identification. The order of each sample packet is as follows:

a. Analytical Results Summary ("CLP SOW288-like" Form I PEST).

b. Copies of herbicide chromatograms.

c. Copies of herbicide chromatograms from second GC column confirmation (if performed).

d. GC integration reports or data system printouts.

e. Exhibit work sheets (including example calculation showing how sample results are calculated using initial calibration and sample responses for at least one sample).

f. UV traces from GPC (if performed).

g. If herbicides are confirmed by GC/MS, the laboratory must submit copies of raw spectra and copies of background-subtracted mass spectra of target compounds that are identified in the sample and corresponding background-subtracted target compound standard mass spectra.

3. Standards Data

a. Analytical Sequence Form -- in chronological order, by GC column, by instrument for all samples and quality control analyses.

b. Initial Calibration Data (Initial Calibration Summary Form [inclusive of retention time windows, calibration factors, %RSDs, %Ds, etc.], herbicide standard chromatograms, and integration reports) -- for each initial calibration associated with SDG in chronological order, by GC column, by instrument. If a curve equation is utilized, the laboratory must provide the curve equation and coefficient of determination.

c. Continuing Calibration Data (Continuing Calibration Summary Form [inclusive of retention time windows, calibration factors, %RSDs, %Ds, etc.], herbicide standard chromatograms, and integration reports) -- for each continuing calibration associated with SDG in chronological order, by GC column, by instrument following the associated initial calibrations.

4. Raw QC Data

a. Blank Data -- in chronological order, by instrument:

i. Target Compound Results ("CLP SOW288-like" Form I PEST).

ii. Herbicide chromatograms and integration reports.

b. Laboratory Control Sample Data:

i. Target Compound Results ("CLP SOW288-like" Form I PEST).

ii. Herbicide chromatograms and integration reports.

c. Matrix Spike Data:

i. Target Compound Results ("CLP SOW288-like" Form I PEST).

ii. Herbicide chromatograms and integration reports.

d. Matrix Spike Duplicate Data:

i. Target Compound Results ("CLP SOW288-like" Form I PEST).

ii. Herbicide chromatograms and integration reports.

e. UV traces from GPC cleanup (if performed).

i. UV traces for the initial calibration standards and blanks. Compound names shall be written or printed over the peaks, or retention times shall be written over the peaks and a separate table listing compounds and retention times shall be provided.

ii. Chromatographs and data system reports for all standards used to quantify compounds in the GPC blanks.

iii. Chromatographs and data system reports for the GPC calibration check solution and all standards used to quantify compounds in the GPC calibration check solution (or used to assess the Aroclor pattern).

K) GC/MS Dioxin/Furan Data

1. Quality Control (QC) Summary

a. Matrix Spike/Matrix Spike Duplicate Summary.

b. Ongoing Precision and Recovery (ORP) Summary.

c. Method Blank Analysis Summary.

2. Sample Data

Sample data shall be arranged in packets consisting of the Analytical Results Summaries followed by the raw data for dioxin/furan samples. These sample packets should then be placed in increasing alphanumeric order by GE sample identification. The order of each sample packet is as follows:

a. Analytical Results Summary.

For each sample including peak retention times, ion ratios, reported concentrations, Estimated Detection Limit (EDL) designation, and internal standard recoveries.

b. Calculation of Toxicity Equivalence.

c. Dioxin/Furan Review Worksheet and Quantitation Report. The quantitation reports must include all information required to reproduce reported positive results and EDL results.

d. Extracted Ion Current Profile (EICP) Chromatograms.

e. Second Column Confirmation Data (if necessary; will include A-9.1.1.K, Section 2, items a, b, c, and d).

f. Exhibit work sheets (including example calibration showing how sample results are calculated using initial calibration and sample responses for at least one sample. The calculations should cover positive results and EDL results).

3. Standards Data

a. Mass spectrometer performance standard data for each calibration associated with the SDG, in chronological order by GC column, by instrument.

b. Window-defining mix and isotope ratio data for each calibration associated with the SDG, in chronological order by GC column, by instrument. The retention time windows must be summarized for reference.

c. Isomer Specificity Test Standard Summary and raw data in chronological order by GC column, by instrument.

d. Initial Calibration Data (Initial Calibration Summary Form, quantitation report, and EICP Chromatograms) for each initial calibration associated with the SDG, in chronological order by GC column, by instrument. If a curve equation is utilized, the laboratory must provide the curve equation and coefficient of determination.

e. Continuing Calibration Data (Continuing Calibration Summary Form, quantitation report, and EICP Chromatograms) for each continuing calibration associated with the SDG, in chronological order, by GC column, by instrument.

4. Raw QC Data

a. Blank Data -- in chronological order, by instrument:

i. Analytical Results Summary.

For each blank including peak retention times, ion ratios, reported concentrations, EDL designation, and internal standard recoveries.

ii. Dioxin/Furan Review Worksheet and Quantitation Report.

iii. EICP Chromatograms.

b. OPR Standard Data:

i. Analytical Results Summary.

For each OPR standard including peak retention times, ion ratios, reported concentrations, EDL designation, and internal standard recoveries.

ii. Dioxin/Furan Review Worksheet and Quantitation Report.

iii. EICP Chromatograms.

c. Matrix Spike Data:

i. Analytical Results Summary.

For each matrix spike including peak retention times, ion ratios, reported concentrations, EDL designation, and internal standard recoveries.

ii. Dioxin/Furan Review Worksheet and Quantitation Report.

iii. EICP Chromatograms.

d. Matrix Spike Duplicate Data:

i. Analytical Results Summary.

For each matrix spike duplicate including peak retention times, ion ratios, reported concentrations, EDL designation, and internal standard recoveries.

ii. Dioxin/Furan Review Worksheet and Quantitation Report.

iii. EICP Chromatograms.

5. GC/MS Instrument Run Logs.

L) Inorganic Data for ICP or ICP/MS

1. Cover Page for the Inorganic Analyses Data Package.

2. Sample Results Summaries (modified CLP SOW390 Form I-INs) -- for all samples in the SDG, arranged in increasing alphanumeric order by GE sample identification.

3. Quality Control and Quarterly Verification of Instrument Parameters Summaries:

a. Initial and Continuing Calibration Verification summaries (modified CLP SOW390 Form II [PART 1]-INs).

b. Detection Limit Standards summaries (if performed, modified CLP SOW390 Form II [PART 2]-INs).

c. Blanks summaries (modified CLP SOW390 Form III-INs).

d. ICP Interference Check Sample summaries (modified CLP SOW390 Form IV-INs).

e. Matrix Spike/Matrix Spike Duplicate Sample Recovery summary (modified CLP SOW390 Form V [PART 1]-IN).

f. Post-Digest Spike Sample Recovery forms (modified CLP SOW390 Form V [PART 2]-IN).

g. Duplicates summary (modified CLP SOW390 Form VI-IN).

h. Laboratory Control Sample summary (modified CLP SOW390 Form VII-IN)

i. Method of Standard Addition Results summary (modified CLP SOW390 Form VIII-IN).

j. ICP Serial Dilution summary (modified CLP SOW390 Form IX-IN).

k. Method Detection Limits (MDL) and Reporting Limits (modified CLP SOW390 Form X-IN).

l. ICP Interelement Correction Factors (if performed, modified CLP SOW390 Form XI [PART 1]-IN).

m. ICP Linear Ranges (if performed, modified CLP SOW390 Form XII-INs).

n. Preparation Logs (modified CLP SOW390 Form XIII-INs).

o. Analytical Run Logs (modified CLP SOW390 Form XIV-INs).

4. ICP/MS Data Package will also include the following additional forms. The forms for ICP analysis listed A-9.1.1.K Sections 1-3 are also required using the SOW1091-LCIN protocol.

a. Linear Range Standard Summary (if performed, modified CLP For IV-LCIN).

b. ICP and ICP/MS Interference Check Sample (modified CLP Form VI-LCIN).

c. ICP/MS Tuning and Response Factor Criteria (modified CLP Form XIV-LCIN).

d. ICP/MS Internal Standards Summary (modified Form XV-LCIN).

5. Raw Data

For each reported value, the contracted laboratories will provide all raw data used to obtain that value. This applies to all required QA/QC measurements, instrument standardization, and all sample analysis results. This statement does not apply to the Quarterly Verifications Parameters submitted as part of each data package (Section A-9.1.1.L, items 3k-3m). Raw data must contain all instrument readouts used for the sample results. Each exposure or instrumental reading must be provided, including those readouts that may fall below the MDL. All AA and ICP instruments must provide a legible hard copy of the direct real-time instrument readout (*i.e.,* strip charts, printer tapes, etc.). A photocopy of the instrument's direct sequential readout must be included. A hard copy of the instrument's direct instrument readout for cyanide must be included if the instrumentation has the capability.

The order of raw data in the data package shall be ICP-AES, ICP/MS, flame AA, furnace AA, mercury, and cyanide. All flame and furnace AA data will be grouped by element.

M) Wet Chemistry/Conventionals Data

The wet chemistry data will be arranged in the following order by individual parameter requested for the samples in the SDG.

1. Analytical Results Summaries -- for all samples in the SDG, arranged in increasing alphanumeric order by GE sample identification.

2. Quality Control Summaries

   a. Initial and Continuing Calibration Verification summaries.

   b. Blanks summaries.

   c. Spike Sample/Spike Duplicate Recovery summary.

   d. Duplicates summary.

   e. Laboratory Control Sample summary.

   f. Analytical Run Logs for instrumental analyses.

3. Raw Data

   For each reported value, the contracted laboratories will provide all raw data (instrument printouts or logbook pages) used to obtain that value. This applies to all required QA/QC measurements, instrument standardization, as well as all sample analysis results. Raw data must contain all instrument readouts/logbooks pages used for the sample results. Each exposure or instrumental reading must be provided, including those readouts/logbook pages that may fall below the quantitation limit. A photocopy of the instrument's direct sequential readout must be included if the instrumentation has the capability.

P) Preparation Logs

1. TCLP Extraction Logs (if TCLP extraction was performed).

2. Volatile Extraction Logs (if medium-level volatile analyses were performed).

3. Semivolatile Extraction Logs.

4. Organochlorine Pesticide/PCB Extraction Logs.

5. Herbicide Extraction Logs.

6. Dioxin/Furan Extraction Logs.

7. Metals Digestion Logs.

8. Wet Chemistry Preparation Logs (by parameter).

A-9.1.3 General Format for Level A Deliverables

A Level A Data Package will be prepared concurrently with each complete Sample Data Package prepared for quality assurance review. The Level A Data Package shall contain data for all samples in one SDG. All Level A Data Packages will be arranged in the following order:

A) Cover Letter/Letter of Transmittal

B) SDG Narrative

This document shall be clearly labeled "SDG Narrative" and shall contain: laboratory name, SDG number, GE sample identifications, laboratory sample numbers, and detailed documentation of any quality control, sample, shipment, and/or analytical problems encountered in processing (preparing and analyzing) the samples reported in the data package. A glossary of qualifier codes used in the SDG must also be provided.

The laboratory must also include any technical and administrative problems encountered, and corrective actions taken. An explanation of all flagged edits (*i.e.,* exhibit edits) on quantitation reports must be included in the SDG Narrative.

Additionally, the SDG Narrative must be signed and dated by the laboratory manager.

C) Field and Internal (Laboratory) Chain-of-Custody Records and Sample Receipt Documentation Log

Copies of both the external and internal Chain-of-Custody Records for all samples within the SDG must be included in the deliverables. A description of the condition and temperature of the samples upon laboratory receipt (*i.e.*, custody seal condition, container status) must be provided for each Chain-of-Custody Record/sample cooler.

D) Analytical Results Summaries, grouped by fraction, and submitted in the same order of fractions as the Level B Deliverables.

A-9.2 Deliverables Reporting Requirements for GC/MS Volatile and Semivolatile Organic Analyses

The laboratory will be required to submit the following information as support documentation for the reported analytical results. The quality control summary forms must include the acceptance criteria (*i.e*., recovery ranges, relative percent difference limits, *etc*.) and spike-added amounts (where applicable). Additionally, the quality control summary forms must indicate any recoveries that are outside of the acceptance criteria. The raw data associated with the samples, blanks, and standards must clearly identify the GE sample identifier, the laboratory sample number, the instrument, the laboratory file number for the analysis, and the peak areas/heights and retention times that correspond to the compounds of interest observed in all analyses reported. If the requirement of a summary form is not applicable to a particular sample, standard, or blank, the requirement should still appear on the form; however, no entry will be necessary on the form for that sample, standard, or blank.

A) 1. An analysis summary of the results for all target compounds for all sample analyses, matrix spike analyses, matrix spike duplicate analyses, laboratory control sample analyses, and method/storage blank analyses must be supplied. The summary must include an entry for each target compound, date(s) and time(s) of analysis, GE sample identification, laboratory sample number, date of sample collection, sample matrix, sample weight, sample percent solids, heated or unheated purge, column type(s), column internal diameter, dilution factor, solid extract volume, solid aliquot volume,

concentration units, and sample results. For semivolatile analyses, date of sample extraction, final extract volume, injection volume, and an indication of whether the GPC cleanup was performed (yes/no) is also required. If positive results below the lowest calibration standard are reported, they must be flagged as estimated ("J") on the analysis summary. "Not-detected" results will be represented by the GE required quantitation limit and a "U" flag. If a compound was detected in a sample as well as in the method blank associated with the sample, the result must be flagged with a "B" on the summary form. Additionally, if a dilution is performed on a sample because a target compound is above the calibration range, then the positive result for the particular compound should be flagged with a "D." If the compound is still above the calibration rage after a dilution is performed on the sample, then the compound should be flagged with an "E."

2. The raw data for the sample analyses, method blank analyses, and storage blank analyses by GC/MS methodologies will include the RICs, mass spectra for all target compounds identified, and quantitation reports for the target compounds and surrogates. The raw data for the matrix spike and matrix spike duplicate analyses will include the RIC and quantitation report for the target compounds. These are required only for Level B Deliverables.

B) A surrogate percent recoveries summary for all of the reported analyses (samples, blanks, *etc*.). The surrogate recovery forms should be segregated by method (*i.e*., high-level solid samples separate from low-level solid samples). The summary form should also include the surrogate recovery limits and the laboratory should flag the compounds that do not meet the recovery limits, on the summary form.

C) A matrix spike/matrix spike duplicate concentration and percent recovery/relative percent difference summary for each matrix spike/matrix spike duplicate pair analyzed. The matrix spike/matrix spike duplicate summary form will indicate the GE identification of the unspiked sample, the MS/MSD sample, the spike concentrations, the matrix, and the concentrations of the compounds present in the unspiked sample and the MS/MSD sample. The summary form should also include the MS/MSD recovery criteria and RPD criterion. The laboratory should flag the compounds that do not meet the criteria. A similar form for the LCS must be included with the deliverables.

D) A method/storage blank summary form for each method/storage blank which identifies the samples associated with each method/storage blank. The date of analysis, time of analysis, file number, and matrix of the method/storage blank must also be reported on the summary form. Storage blanks are only required for volatiles analysis.

E) 1. A GC/MS tuning summary which summarizes the percent abundances for the mass ions of interest and the acceptance criteria for the mass ions. Additionally, the summary must include a list of the sample and QC sample analyses (sample names, file numbers, and dates and times of analysis) associated with the GC/MS tune. The summary should indicate the instrument identification, date and time of analysis, column type, diameter of the column, and the type of purge (heated or unheated for volatiles) used to analyze the samples.

2. The raw data for the GC/MS tuning summary, consisting of a summary of the mass ion abundances and a mass spectral representation of the tuning peak.

F) 1. For the internal standard calibration method, an initial calibration summary for each initial calibration performed, summarizing all of the relative response factors for each calibration standard, the average relative response factor, and the relative standard deviation among the relative response factors. If a calibration curve equation is utilized, the laboratory must summarize the curve equation and the coefficient of determination. Additionally, the summary should indicate the compounds that must meet a minimum relative response factor or a maximum relative standard deviation criterion and the compounds that did not meet the acceptance criteria. The summary should indicate the instrument identification, the file identifications of the analyses, the dates and times of calibration commencement and completion, column type, diameter of the column, and the type of purge (heated or unheated for volatiles) used to analyze the samples.

2. The raw data for the initial calibration, consisting of the reconstructed ion chromatogram and the raw quantitation report for each calibration standard. This is a requirement for the Level B Deliverables only.

G) 1. For the internal standard calibration method, a continuing calibration summary for each continuing calibration standard analyzed, summarizing the average relative response factors of the initial calibration associated with the continuing calibration standard, the relative response factors of the continuing calibration standard, and the percent differences between the

average relative response factors of the initial calibration and the relative response factors of the continuing calibration. If calibration curve equations are utilized the laboratory must summarize the true concentration, observed concentration, and the percent drift. Additionally, the summary must indicate the compounds that are subject to a minimum relative response factor criterion, the compounds that are subject to a maximum percent difference criterion, and the compounds that did not meet the acceptance criteria. The summary should indicate the instrument identification, the date of the initial calibration, the date and time of analysis, column type, diameter of the column, and the type of purge (heated or unheated for volatiles) used to analyze the samples.

2. The raw data for the continuing calibration, consisting of the reconstructed ion chromatogram and the raw quantitation report for each calibration standard. This is a requirement only for the Level B Deliverables.

H) An internal standard area counts summary, containing a summary of the area counts and retention times for the internal standards for a continuing calibration. The summary must indicate the acceptance windows for the internal standard retention times and area counts. This summary must supply a comparison of the continuing calibration internal standards to the mid-level initial calibration internal standards. Additionally, the summary must include a listing of the internal standard retention times and area counts for all of the samples, method blanks, matrix spikes, and matrix spike duplicates associated with the continuing calibration standard.

I) A copy of all of the extraction log information for semivolatiles is required. At a minimum, the extraction information must include the date the extraction was

started, the date the extraction was completed, the initial sample weight or volume, final extraction volume, laboratory sample number, the amount and concentration of surrogate spike added, and the amount and concentration of matrix spike solution added. Additionally, the extraction log should indicate if a cleanup procedure was performed on the sample. If a medium-level extraction was performed for the volatiles analysis, all extraction logs for this analysis will be required. For volatile organics analyses that require weighing sample aliquots in the field, copies of the field measurement documentation will be included in this section.

A-9.3 Deliverables Reporting Requirements for Organochlorine Pesticide, PCB, and Herbicide Analysis

The laboratory will be required to submit the following information as support documentation for the reported analytical results. The quality control summary forms must include the acceptance criteria (i.e., recovery ranges, relative percent difference limits, etc.) and spike-added amounts (where applicable). Additionally, the quality control summary forms must indicate any recoveries that are outside of the acceptance criteria. The raw data associated with the samples, blanks, and standards must clearly identify the GE sample identification, the laboratory sample number, the instrument, the laboratory file number for the analysis, and the peak areas/heights and retention times that correspond to the compounds of interest observed in all analyses reported. If the requirement of a summary form is not applicable to a particular sample, standard or blank, the requirement should still appear on the form; however, no entry will be necessary on the form for that requirement.

A) 1. An analysis summary of the concentrations of all target compounds for all sample analyses, matrix spike analyses, matrix spike duplicate analyses, and blank analyses. The blank analyses must consist of all of the extraction

(method) blank analyses, injection blank analyses, and any blanks associated with cleanup procedures. The summary must include dates and times of analysis, GE sample identifications, laboratory sample numbers, dates of sample collection, date of sample receipt, dates of sample extraction, sample matrices, sample weights or volumes, sample percent solids, column types, column internal diameters, dilution factors, initial extract volumes/weights, final extract volumes, concentration units, the type of cleanup performed, and sample results. If positive results below the lowest calibration standard are reported, they must be flagged as estimated ("J") on the analysis summary. "Not-detected" results will be represented by the GE required quantitation limit and a "U" flag. If a compound was detected in a sample as well as in the method blank associated with the sample, the result must be flagged with a "B" on the summary form. Additionally, if a dilution is performed on a sample because a target compound is above the calibration range then the positive result for the particular compound should be flagged with a "D." If the compound is still above the calibration range after a dilution is performed on the sample, then the positive result for the compound should be flagged with an "E."

2. The raw data for the sample analyses, matrix spike analyses, matrix spike duplicate analyses, and blank analyses, consisting of the chromatograms indicating the surrogate peaks and target compound peaks and quantitation reports for the target compounds and surrogates. This is a requirement only for the Level B Deliverables.

B) A surrogate percent recovery summary for all of the reported analyses (samples, blanks, *etc*.). The surrogate recovery forms should be segregated by matrix and

method (*i.e.*, medium-level solid samples separate from low-level solid samples). The summary form should also include the surrogate recovery limits and the laboratory should flag the compounds that do not meet the recovery limits on the summary form.

C) A matrix spike/matrix spike duplicate concentration and percent recovery/relative percent difference summary for each matrix spike/matrix spike duplicate pair analyzed. The matrix spike/matrix spike duplicate summary form will indicate the GE identification of the unspiked sample, the MS/MSD sample, the spike concentrations, the matrix, and the concentrations of the compounds present in the unspiked sample and the MS/MSD sample. The summary form should also include the MS/MSD recovery criteria and RPD criterion. The laboratory should flag the compounds that do not meet the criteria. A similar form for the LCS should be included with the deliverables.

D) A method blank summary form for each method blank, identifying the samples associated with each method blank. The date, time, lab file number, and matrix of the method blank must also be reported on the summary form.

E) Initial Calibration Data: A summary of the initial calibration retention times, mean retention time, and a retention time window for all target compounds and surrogates must be provided for all initial calibrations. A second summary of the initial calibration standard calibration factors, average calibration factors, and relative standard deviations for all target compounds and surrogates must also be provided for all initial calibrations. If a calibration curve equations is utilized the laboratory must supply the curve equation and the coefficient of determination. Both summaries should include the SDG number, instrument identification, GC column

type and diameter, date(s) of analysis, the concentration level for each initial calibration standard (as a multiplication factor of the low calibration standard), and the acceptance limit for the relative standard deviation. Copies of the pesticide, herbicide, and PCB standard chromatograms and integration reports associated with summaries should immediately follow the summary (only for the Level B Deliverables). Each initial calibration associated with the SDG must be presented in chronological order, by GC column and by instrument.

F) Continuing Calibration Data: A summary of the observed retention times, calculated compound concentrations, true concentrations, percent differences, and retention time window from the initial calibration (or from the daily retention time window update) must be provided for all continuing calibration standards. If calibration curve equations are utilized the laboratory must summarize the true concentration, observed concentration, and the percent drift. The summary should list the SDG number, GC column type and diameter, date and time of analysis, laboratory sample number, initial calibration dates, and acceptance limits. Copies of the pesticide, herbicide, and PCB standard chromatograms and integration reports associated with summaries should immediately follow the summary (only for the Level B Deliverable). Each continuing calibration associated with an SDG must be presented in chronological order, by GC column and by instrument.

G) 4,4′-DDT and Endrin Breakdown Data (organochlorine pesticides only): A summary of the observed 4,4′-DDT, endrin, and combined percent breakdowns must be presented for each breakdown check performed. (Alternatively, if this data is obtained from a continuing calibration standard rather than a specific breakdown standard, this information may be reported on the associated continuing calibration

summary form.) The summary should list the SDG number, GC column type and diameter, date and time of analysis, laboratory sample number, initial calibration dates, and acceptance limits. Copies of the pesticide/PCB standard chromatograms and integration reports associated with summaries should immediately follow the summary (only for the Level B Deliverables). Each breakdown summary associated with an SDG must be presented in chronological order, by GC column and by instrument.

H) A summary of the analytical sequence for each column and instrument used for the analysis of the project samples. The summary must contain the GC column number, the internal diameter of the column, initial calibration dates associated with the sequence, the instrument identification, the mean retention time(s) for the surrogate(s) utilized, a listing of the GE sample names, the laboratory sample numbers, dates and times of analysis, and the retention times for the surrogate(s). The summary should also indicate the retention time window for all surrogates used and any surrogate retention times that do not meet the acceptance criterion. The summary must contain all of the analyses for the samples, blanks, initial calibration standards, and continuing calibration standards associated with the sequence. All sequences will begin with an initial calibration and will terminate with a continuing calibration or breakdown check standard that meets all acceptance criteria.

I) When a GPC cleanup procedure is required for the samples, a summary for each check standard associated with the GPC calibration. The summary must contain the GPC column identification, the calibration date of the GPC column, the GC column(s) used for the analysis of the standard, the GC column internal diameter, the theoretical concentrations of the compounds in the GPC standard, the observed concentrations of the GPC standard, the percent recovery for each compound in the

GPC standard, the GE sample identification, laboratory sample number, and the date(s) of analysis for all samples associated with the GPC standard. The limits for each compound in the GPC standard should be listed on the summary form. The laboratory should flag any compound if the percent recovery was not within the control limits.

J) When a Florisil® cartridge cleanup procedure is required for the samples, a summary for each check standard associated with a Florisil® cartridge lot. The summary must contain the Florisil® cartridge lot number, the date of analysis of the Florisil® cartridge check standard, the GC column(s) used for the analysis of the standard, the GC column internal diameter(s), the theoretical concentrations of the compounds in the Florisil® cartridge check standard, the observed concentrations of the Florisil® cartridge check standard, the percent recovery for each compound in the Florisil® cartridge check standard, the GE sample identifications, the laboratory sample number, and the date(s) of analysis for all samples in the data deliverable associated with each lot of Florisil® cartridges.

K) Second column confirmation may be performed for all pesticide, PCB, and herbicide analyses when there is a positive result reported for a project sample. When the laboratory performs a dual column quantitative analysis for organochlorine pesticides, PCBs, and herbicides, a summary of the identified compounds and observed concentrations for the two columns utilized for sample analyses is required. The summary must contain the GE sample identification, the laboratory sample number, the dates and times of analysis, the instruments used for analysis, the GC columns, the GC column internal diameters, the retention time windows for each peak used to quantitate the compound, the observed retention time for each

peak used to quantitate the compound, the calculated concentration for each peak used, the mean concentration for each column for each compound identified, and the percent difference between the mean concentrations calculated for each column.

If the percent difference between the results for the analyte from the two GC columns is greater than 40% for the analysis, then the higher of the two values is reported and flagged with a "P." Finally, the "C" flag is used when the identification of a pesticide result is confirmed by GC/MS.

A-9.4 Deliverables Reporting Requirements for Dioxin/Furan Analyses

The laboratory will be required to submit the following information as support documentation for the reported analytical results. The quality control summary forms must include the acceptance criteria (*i.e*., recovery ranges, relative percent difference limits, *etc*.) and spike-added amounts (where applicable). Additionally, the quality control summary forms must indicate any recoveries that are outside of the acceptance criteria. The raw data associated with the samples, blanks, and standards must clearly identify the GE sample identifier, the laboratory sample number, the instrument, the laboratory file number for the analysis, and the peak areas/heights and retention times that correspond to the compounds of interest observed in all analyses reported. The raw data must provide all information necessary to reproduce all reported positive and EDL results. If the requirement of a summary form is not applicable to a particular sample, standard, or blank, the requirement should still appear on the form; however, no entry will be necessary on the form for that requirement.

A) 1. An analysis summary of the results for all target compounds for all sample analyses, second column confirmation analyses, matrix spike analyses, ORP

standard analyses, and method blank analyses must be supplied. The summary must include an entry for each target 2,3,7,8-substituted compound and total homologue concentrations, date(s) and time(s) of analysis, GE sample identification, laboratory sample number, date of sample collection, date of sample preparation, sample matrix, sample weight, sample percent solids, column type(s), column internal diameter(s), dilution factor, concentrated extract volume, concentration units, peak retention times, isotope ratios, and sample results. If positive results below the lowest calibration standard are reported, they must be flagged as estimated ("J") on the analysis summary. "Not-detected" results will be represented by the EDL and a "U" flag. If a compound was detected in a sample as well as in the method blank associated with the sample, the result must be flagged with a "B" on the summary form. Additionally, if a dilution is performed on a sample because a target compound is above the calibration range, then the positive result for the particular compound should be flagged with a "D". If the compound is still above the calibration rage after a dilution is performed on the sample, the positive result for the compound should be flagged with an "E".

2. The raw data for the sample analyses and method blank analyses by GC/MS methodologies, consisting of the EICP, quantitation reports for the target compounds, the associated areas or height for each peak within the established retention time window, and all other information required to reproduce all reported positive and EDL results. The raw data for the matrix spike and matrix spike duplicate analyses will include the EICP chromatogram and quantitation report for the target compounds.

B) A matrix spike concentration and percent recovery summary for each matrix spike analyzed is required. The matrix spike summary form will indicate the GE identification of the unspiked sample, the sample, the matrix, and the concentrations of the compounds present in the unspiked and spiked sample. The summary form should also include the MS recovery criteria. The laboratory should mark the compounds that do not meet the specified criteria. A similar form for the OPR standard should be included with the deliverables.

C) A method blank summary form for each method blank that identifies the samples associated with each method blank. The date of extraction, date of analysis, time of analysis, lab file number, sample weight, and matrix of the method blank must also be reported on the summary form.

D) A mass spectrometer performance summary for each mass spectrometer performance standard analyzed should identify the sample number, lab file identification, date and time of analysis, instrument identification, GC column identification, and static resolving power.

E) A window defining mix summary form for each window defining analysis should identify the sample number, lab file identification, date and time of analysis, instrument identification, and GC column identification. This form should include the retention time of the first eluting and last eluting isomer for each congener group.

F) An isomer specificity test standard summary should identify the sample number, file number, instrument ID, date and time of analysis, the GC column and instrument identification, and the percent valley determination between $^{13}C_{12}$-2,3,7,8-TCDD and $^{13}C_{12}$-1,2,3,4-TCDD. In addition, if second column confirmation is required,

percent valley for 2,3,7,8-TCDD and the closest isomers should be calculated and reported.

G) A summary of the analytical sequence for each column and instrument used for the analysis of the project samples. The summary must contain the GC column number, the internal diameter of the column, initial calibration dates associated with the sequence, the instrument identification, a listing of the GE sample names, the laboratory sample numbers, and dates and times of analysis. The summary must contain all of the analyses for the samples, blanks, initial calibration standards, and the continuing calibration standards associated with the sequence.

H) 1. An initial calibration summary for each initial calibration performed, summarizing all of the relative response factors for each calibration standard, the average relative response factor, and the relative standard deviation among the relative response factors. If calibration curve equations are utilized, the laboratory must supply the curve equation and coefficient of determination. Additionally, the summary should indicate maximum relative standard deviation and minimum relative response factor criteria as well as the compounds that did not meet the acceptance criteria. The summary should indicate the instrument identification, the dates and times of calibration commencement and completion, column type, and diameter of the column.

2. The raw data for the initial calibration, consisting of the EICPs and the raw quantitation report for each calibration standard.

I) 1. A continuing calibration summary for each continuing calibration standard analyzed, summarizing the average relative response factors of the initial calibration associated with the continuing calibration standard, the relative response factors of the continuing calibration standard, and the percent differences between the average relative response factors of the initial calibration and the relative response factors of the continuing calibration, and the isotope ratios and retention times. If calibration curve equations are utilized the laboratory must summarize the true concentration, observed concentration, and the percent drift. Additionally, the summary must indicate the compounds that are subject to a minimum relative response factor criterion, the compounds that are subject to a maximum percent difference criterion, and the compounds that did not meet the acceptance criteria. The summary should indicate the instrument identification, the date of the initial calibration, the date and time of analysis, column type, and diameter of the column.

2. The raw data for the continuing calibration, consisting of the EICPs and the raw quantitation report for each calibration standard.

A-9.5 Deliverables Reporting Requirements for Inorganic Analyses

The laboratory will be required to submit the following information as support documentation for the reported analytical results. The quality control summary forms must include the acceptance criteria (*i.e.,* recovery ranges, relative percent difference limits, *etc*.) and spike-added amounts (where applicable). Additionally, the quality control summary forms must indicate any quality control results that are outside the acceptance criteria. All instrument raw data printouts for the points discussed below must be provided in an orderly

fashion. This applies to all required QA/QC measurements, and instrument standardization, as well as sample analysis results. Additionally, all associated extraction, digestion, and distillation logs must be supplied. The order of the raw data in the data package shall be ICP-AES, ICP/MS, flame AA (if performed), furnace AA (if performed), and mercury. All flame and furnace AA data shall be grouped by element. All raw data shall be grouped by analysis date for all analytical results.

A) 1. A sample reference list for all samples present in an SDG. This reference list must summarize and correlate the laboratory sample number, the GE designated sample identification, and any laboratory code (*i.e.*, truncation of GE designated sample number by the laboratory) for each sample in an SDG.

2. A Table of Contents listing page numbers associated with information such as:

a. Methodology Summary

b. Case Narrative

c. Sample Results

d. Quality Control Data

e. Verification of Instrument Parameters

f. Preparation and Analysis Logs

g. Raw Data, including but not limited to:

i. ICP-AES, ICP/MS, Flame AA, GFAA, and Mercury Data

ii. Digestion Logs

iii. Confirmation Data

h. Chain-of-Custody Records

B) Analysis summaries of the concentrations of all target analytes for all sample analyses. The summary must include the GE designated sample number, the laboratory sample number, date of sample collection, date of sample receipt, sample matrix, sample percent solids, concentration units, sample results, data qualifier codes, analysis method codes, description of sample before and after analysis, and any comments relating to the sample.

C) A summary of the initial and continuing calibration verifications for each calibration performed. This summary will include the concentrations observed as well as the true value of the analyte in the initial and continuing calibrations. A percent recovery will be summarized based on the observed and true values for each analyte.

D) A summary of the Detection Limit (DL) standard analyses for both Atomic Absorption (AA) and Inductively Coupled Plasma (ICP) analyses. This summary will include the concentrations observed as well as the true value of the analyte in the DL standard. A percent recovery will be summarized based on the observed and true values for each analyte.

E) A summary of the initial and continuing laboratory blank analyses for each calibration performed. This summary will include the concentrations (positive or negative) observed of any analyte in the initial and continuing blank analyses at values greater than the MDL. The summary should also include the concentrations of any analyte observed in the laboratory preparation blank associated with each calibration sequence performed by the laboratory.

F) A summary of the ICP interference check sample analysis for each analytical sequence performed. This form will summarize the true and found values (positive, negative, or zero) of all analytes present in Solutions A and AB of the ICP interference check sample analysis. This form will also summarize the percent recoveries of the analytes/interferences present in the standards.

G) A summary of the pre-digestion matrix spike analysis. This form will summarize the percent recovery control limit for each analyte. Also, the sample result, the spike sample result, and the spike-added amount must be summarized on this form for all parameters analyzed. The laboratory-calculated percent recovery as well as the laboratory qualifier stating whether the calculated percent recovery was within control limits must also be summarized on this form.

H) A summary of the post-digestion matrix spike analysis. This form will require the same information described in item G.

I) A summary of the laboratory duplicate analysis. This form will summarize the percent differences observed between the sample and laboratory duplicate analyses. The appropriate control limits must be specified by the laboratory, and a summary of

the sample and laboratory duplicate analyses must be provided. The percent solids for the sample and the duplicate sample should be included on the summary form.

J) A summary of the Laboratory Control Sample (LCS) analysis. This form will summarize the percent recovery, control limits, and true and found values for the solid sample analyses.

K) A summary of any required Method of Standard Additions (MSA) determinations. This form will summarize the concentrations and absorbencies of all samples and analytes that require analysis by MSA. The correlation coefficient for the MSA analysis will be calculated and summarized on this form. Also, the sample concentration determined from the MSA determination will be summarized on this form.

L) A summary of the ICP Serial Dilution analyses performed by the laboratory. This summary will show the result of the initial sample analysis (in aqueous units, as observed from the raw data), the result of the five-fold serial dilution analysis, and the percent difference between the two analyses.

M) The summaries necessary for the verification of instrument parameters. These include an Method Detection Limit and Reporting Limit Summary, an ICP Interelement Correction Factor Summary (if performed) for each ICP used for analysis, and an ICP Linear Range Summary (if performed) for each ICP used for analysis.

N) The analysis log summaries. These include a Sample Preparation log that provides the sample identification; the preparation date; the sample weight (in grams) used;

and the digestion volume (in mL) used and an Analysis Run Log that provides the instrument identification, the sample identification, any dilution factors employed in the analysis, the date and time of analysis, the method of analysis, and the parameters analyzed. Additionally, the GFAA post-digestion analytical spike sample recoveries are listed on the Analysis Run Log.

A-9.6 Deliverables Reporting Requirements for Wet Chemistry/Conventionals Analysis

The laboratory will be required to submit the information detailed in Sections A-9.5 A) -C), A-9.5, E) and A-9.5, G) - J) and A-9.5-N as support documentation for the reported analytical results. The quality control summary forms must include the acceptance criteria (*i.e*., recovery ranges, relative percent difference limits, *etc*.) and spike-added amounts (where applicable). Additionally, the quality control summary forms must indicate any quality control results that are outside the acceptance criteria. All instrument raw data printouts for the points discussed in the above mentioned sections must be provided in an orderly fashion. This applies to all required QA/QC measurements, and instrument standardization, as well as sample analysis results. Additionally, a direct sequential readout must be included if the instrument has the capability.

APPENDIX 10

METHOD: ASTM D422
STANDARD OPERATING PROCEDURE
FOR PARTICLE SIZE ANALYSIS OF SOILS
Applicable Matrix or Matrices: Soil, Sediment, Sludge
Standard Compound List and Reporting Limits: NA

Approvals and Signatures

Laboratory Director:	Christopher A. Ouellette	Date: 5-12-00
QA Manager:	Kim B. Watson	Date: 5-12-00
Inorganics Technical Director:	Kristine A. Dusablon	Date: 5-12-00
Geotechnical Supervisor:	Jeffrey R. McMahon	Date: 5/12/00

1.0 SCOPE AND APPLICATION

1.1 This method determines the particle size distribution in soil. Particles greater than 75um (gravels to fine sands) are determined by sieving and particles less than 75um (silts and clays) are determined by sedimentation using an hydrometer.

1.2 Minimum quantity of sample depends on subsequent analyses to be performed. Typical range is 150 to 350 grams of dry soil. Larger amounts (from 500 to 5000 grams) are specified for particle size analysis of soils with appreciable gravel component.

1.3 This preparation is amenable to samples containing sand, silt, clay and gravel.

2.0 SUMMARY OF METHOD

2.1 Soils for particle size analysis are prepared according to ASTM D421 or D2217. The soils are sieved in two steps. The particles greater than 2.00mm (retained on the No. 10 sieve) are sieved after the soil has been prepared. A portion of the soil passing the No. 10 sieve is prepared for hydrometer measurements. Seven hydrometer readings are made over a 24 hour time frame. The soil in the hydrometer is rinsed on a No. 200 (75 um) sieve and dried for sieve analysis of material less than 2.00mm (No. 10 sieve). Calculations are made to determine the percent finer of soil for each sieve and hydrometer reading. These calculations are dependent on percent solid, which is determined during the drying process, and the specific gravity that is assumed to be 2.65 (unless separate analysis is requested for specific gravity).

3.0 DEFINITIONS

N/A

4.0 INTERFERENCES

N/A

5.0 SAFETY

5.1 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous materials or procedures.

5.2 STL Burlington maintains a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. Material Safety Data Sheets (MSDS) are made available to all personnel involved in the chemical analysis. STL Burlington also has a written environmental health and safety plan.

5.3 Please note chemicals that have the potential to be highly toxic or hazardous, the appropriate MSDS must be reviewed by the employee before handling the chemical.

6.0 EQUIPMENT AND SUPPLIES

6.1 Balance sensitive to 0.01 grams

6.2 Mixer and dispersion cup

6.3 1000 ml sedimentation cylinder

6.4 Soil test hydrometer meeting specification E 100

6.5 Mortar and rubber tipped pestle for breaking up soil aggregates

6.6 Sieves of the following size:

3.0 in (75.00mm)	No. 20 (850.0um)
2.0 in (50.00mm)	No. 40 (425um)
1.5 in (37.50mm)	No. 60 (250.0um)
1.0 in (25.00mm)	No. 80 (180.0um)
3/4 in (19.00mm)	No. 100 (150.0um)
3/8 in (9.50mm)	No. 200 (75.0um)
No. 4 (4.75mm)	
No. 10 (2.00mm)	

6.7 Oven with temperature range of 60° C to 110° C

6.8 Thermometer accurate to 0.5° C

6.9 Timer with second hand and capable of counting up to 25 hours

6.10 Mixing utensils, metal and bristle brushes for sample recovery.

6.11 Rototap machine

7.0 REAGENTS AND STANDARDS

7.1 Sodium Hexametaphosphate (dispersion reagent)

8.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

8.1 Typical sample of 150 to 350 grams is used for analysis. Larger amounts (from 500 to 5000 grams) are specified for particle size analysis of soils with appreciable gravel component. The sample container must remained sealed to maintain natural water content.

8.2 There are no holding time requirements.

9.0 QUALITY CONTROL

9.1 Check balance daily with Class S weight, yearly manufacturer calibration.

9.2 Oven temperature is checked daily prior to start of work.

9.3 Thermometer is checked against similar or more accurate temperature device.

9.4 Duplicate samples are recommended every 20 samples.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Sieves calibrated twice a year using the National Bureau of Standard, Certificate of Calibration, standard reference materials 1017a, 1018a and 1019a calibrated glass beads.

10.2 Hydrometers are calibrated twice a year, and checked prior to each use.

10.3 Thermometer calibrated against NIST certified thermometer.

11.0 PROCEDURE

11.1 Large Sieve (dry): The soil retained on the No. 10 sieve is used in this step. Rinse the particles on a No. 10 sieve and then place the material in an oven until dry.

Large Sieve (wet): Take the equivalent of 200 grams of dry soil (use the percent solid table). Place soil on a No. 10 sieve and wash the soil. Take the soil retained on the No. 10 sieve and place in an oven until dry.

11.1.1 Record the weights of the sieves greater than No. 10. Take the dry soil and pour into the sieve stack. Place the sieve stack on the Rototap machine and shake sample for ten minutes.

11.1.2 Weigh and record the contents of each sieve.

11.1.3 Record the maximum particle size. Determine the hardness of the particles by dropping a hammer on the particle from a height or approximately one foot. Record the hardness as hard, soft or brittle. Save the soil particles.

11.2 Hydroscopic Moisture (dry prep. only): The soil passing the No. 10 sieve is used in this step. Take a small tin, label it and record the weight. Place approximately 10 to 15 grams of soil in the tin. Place the tin in the oven at 110°C for at least 16 hours. Remove the tin and record the weight.

11.3 Hydrometer Test: The soil passing the No. 10 sieve is used in this step.

11.3.1 Sample Preparation:

- Dry Prep: Tare a 250 ml beaker. Place and record approximately 50 grams for silt or clay particles or 100 grams for same particles into the beaker. Add 125 ml of a 40g/l sodium Hexametaphosphate solution to sample and allow to soak overnight

  Wet Prep: Tare a 500 ml beaker. Place and record the dry equivalent (use the percent solid table) of approximately 50 grams for silt or clay particles into the beaker. Add 125 ml of a 40g/l sodium Hexametaphosphate solution to sample.

- Dry Prep: Rinse the sample with DI water into a dispersion cup. Fill the cup to the halfway mark with DI water and place cup on the blender. Mix sample for approximately one minute. Pour content of cup into a 1000 ml sedimentation cylinder. Rinse cup with DI water to wash all the sample into cylinder. Fill the cylinder to the 1000 ml line and cover with a sheet of paraffin wax.

  Wet Prep: Rinse the sample with DI water into a dispersion cup. Fill the cup to the halfway mark with DI water and place cup on the blender. Mix sample for approximately five minutes. Pour content of cup through a No. 10 sieve into a 1000 ml flask. Rinse cup with DI water to wash all the sample into the flask. Fill the flask to the 1000

ml line and cover the flask with a sheet of paraffin wax. Take the material on the No. 10 sieve, dry it in the oven and record the weight.

11.3.2 After preparing up to 12 flasks, begin setup for hydrometer readings. The following paperwork is needed: hydrometer data sheet, hydrometer reading table, and temperature table if conversion from Fahrenheit to Celsius is necessary. Initiate timer to indicate the elapsed time, counting up from zero. Check readings of hydrometer and temperature probe in a DI water rinse bath. Get the rubber stopper to shake flask and prepare staging and test areas.

11.3.3 Initiate timer to indicate the elapsed time. The hydrometer reading table is used to perform activities as indicated (shake, place or read) for each 1000 ml cylinder.

A reading consists of inserting the hydrometer gently into the cylinder, (after the cylinder has been shaken for 1 minute), about 20 seconds before the actual reading. Read the hydrometer to the nearest 0.0005 at the top of the meniscus. Remove the hydrometer and insert a temperature sensor into the cylinder to the depth to which the hydrometer reached. Read the temperature meter to the nearest 0.1°C and remove the temperature sensor. The hydrometers and temperature sensor are rinsed in a DI bath between each reading.

After each cylinder is read, the hydrometer reading, temperature, and time (from table) is entered on the hydrometer data sheet at the corresponding cylinder (test) number and time portion on the data sheet; deviations from the table schedule are noted on the sheet. The readings are taken at 2, 5 and 15 minute and at 30, 60, 240 and 1440 minutes.

11.4 Small Sieve: Soils from the hydrometer test are rinsed on the No. 200 sieve. The soil retained on the No. 200 sieve is placed in an oven and dried over night.

11.4.1 Record the weights of the sieves used between No. 10 and No. 200. Take the dry soil and pour into the sieve stack. Place the sieve stack on the Rototap machine and shake sample for ten minutes.

11.4.2 Weigh and record the contents of each sieve.

12.0 CALCULATIONS

12.1 Percent Solids (PS) and Hydroscopic Moisture Correction Factor (HMCF)

12.1.1 HMCF is used for air dried samples (dry prep.)

*HMCF = (pan and baked sample - pan)/(pan and dry sample -pan)*100*

12.1.2 Wet Method:

*PS = (pan and dry sample- pan)/(pan and wet sample - pan)*100*

Dry Method:

*PS = HMCF * (pan and dry sample -pan)/(pan and wet sample - pan)* 100*

12.2 Sample Used (SU):

12.2.1 Wet Method:

SU = (pan and wet sample - pan) PS*

Note: for hydrometer SU, subtract the dry weight of any material retained on the No. 10 sieve.

12.2.2 Dry Method:

*SU = ((pan and dry sample - pan) - (pan and non-soil material - pan)) * HMCF*

12.3 Sieve Analysis (Percent Finer = PF)

12.3.1 Large Sieves:

- 3 inch: PF = 100-100* (Sieve and Sample (3 inch) - Sieve (3 inch))/SU
- 2 inch: PF = PF (3 inch) - 100*(Sieve and Sample (2 inch) - Sieve (2 inch))/SU and so on through the #10 Sieve.

12.3.2 Small Sieves

- #20: PF = PF(#10) - 100*(mass passing #10/sample mass (Hyd))*(sieve and sample (#20) - sieve(#20))/sample used
- #40: PF = PF (#20) - 100*(mass passing #10/sample mass (Hyd))*(sieve and sample (#40) - sieve (#40))/sample used and so on up through #10 sieve.

12.4 Hydrometer Analysis

12.4.1 Particle size, Micron

*1000*sqrt [930*viscosity/980*(SG-1))*(effective depth/time)]*

Viscosity at sample temperature, poises

- Effective Depth, cm = 16.29-264.5*(actual Hydrometer reading - 1) above equation for effective depth based on equation found with table 2 in method, in which 16.29 = 0.5*(14.0-67.0/27.8)+10.5 and 264.5 = (10.5-2.3)/0.031
- Time, minutes = Time of hydrometer reading from beginning of sedimentation

Sqrt - square root
SG - Specific Gravity of soil
Viscosity - is the resistance of a liquid to flow

12.4.2 Percent Finer (PF):

PF = Constant(actual hydrometer reading - hydrometer correction factor - 1)*

Where:
Constant = (100,00/W)*SG/(SG-1)
W = (Total sample used *sample used for hydrometer analysis*HMCF)/Amount of total sample passing #10 sieve
Hydrometer Correction = slope*sample temperature + Intercept
Slope = ((low temp. reading -1)-(high temp. reading -1)/(low temp. - high temp.))
Intercept = (low temp. reading -1) - (low temp. * slope)

13.0 METHOD PERFORMANCE

N/A

14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The USEPA has

established a prevention hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the agency recommends recycling as the next best option.

14.2 The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

14.3 For information about pollution prevention that may be applicable to laboratories and research institutions, consult "Less is Better: Laboratory Chemical Management for Waste Reduction", available from the American Chemical Society's Department of Government Regulations and Science Policy, 1155 16th Street N.W., Washington, D.C. 20036; (202) 872-4477.

15.0 DATA ASSESSMENT AND CRITERIA AND CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

15.1 Data is initially reviewed by the analyst in the lab. Following this, the data is secondarily reviewed by QC personnel before being put into its final data package form (where the data is thirdly reviewed before being sent to the client).

15.2 Data that is out of control is marked as such and slated for re-analysis. Any corrective action undertaken is documented on a corrective action form (detailing the client information, problem, investigation findings and solution). This form is kept together with the project.

16.0 CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

16.1 Generally, any data that is out of control is considered unusable. There are, however, cases in which laboratory supervisor will be made aware of the issue and, if the data is used, it will be thoroughly narrative noted.

17.0 WASTE MANAGEMENT

17.1 The USEPA requires that laboratory waste management practices conducted be consistent with all applicable rules and regulations. Excess reagents, samples, and

method process wastes should be characterized and disposed of in an acceptable manner. The Agency urges laboratories to protect the air, water and land by minimizing and controlling all releases from hoods, and bench operations, complying with the letter and spirit of any waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management consult the "Waste Management Manual for Laboratory Personnel", available from the American Chemical Society at the address listed in Section 14.3.

18.0 REFERENCES

18.1 Annual Book of ASTM Standards, volume 04.08 Soil and Rock (I): D 420 - D4914, Section 4, Construction edition; American Society for Testing and Materials, Philadelphia, Pa., 1994.

19.0 TABLES, DIAGRAMS, FLOWCHARTS AND VALIDATION FORMS